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CALCIUM INORGANIC ELECTROLYTE BATTERY DEVELOPMENT. (U)

CALCIUM INORGANIC ELECTROLYTE BATTERY
MAR 82 D CARR, E HEATON, B HIGGINS

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CALCIUM INORGANIC ELECTROLYTE BATTERY DEVELOPMENT

Eagle-Picher Industries, Inc.
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March 1982

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WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

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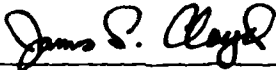
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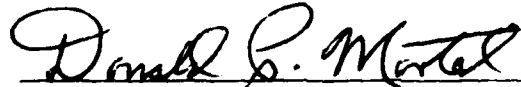
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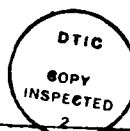
3. Intermittent discharging characterization.
4. Reversed polarity abuse.

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SECTION I

GENERAL INTRODUCTION

Lithium/thionyl chloride electrochemical cells have developed into a viable 400 to 600 watt-hours per kilogram (180 to 270 watt-hours per pound) system in cell sizes up to 17,000 ampere hours. However, the system has suffered from safety problems, in part due to the low melting point of lithium. Calcium has a much higher melting point and when used in a cell with thionyl chloride, is capable of 5.93 watt-hours per cubic centimeter (97.2 watt-hours per cubic inch) and has a working potential of three volts.

The higher melting point of calcium significantly reduced the danger of fires due to heat producing situations such as short circuits and punctures; however, more violent abuse such as incineration and penetration by bullets has produced intense fires.

Development of a thicker cathode using a current collector of foamed Nickel 200 led to a low surface area cell design which further reduced the danger during abuse situations. No condition of abuse has been found which produced a hazardous response from low surface area calcium/thionyl chloride cells as large as 600 ampere-hours. Previous discharge tests on low surface area cells yielded sporadic successes. The failures were believed due to three factors. 1) Two different thionyl chloride electrolyte solutions were used. One of these produced 20%-30% less capacity than the other. 2) The fabrication method used resulted in a cathode surface which tended to short circuit through the separator. We believe these short circuits

GENERAL INTRODUCTION (continued)

had burned themselves out, but not before they reduced the overall cell capacity.

3) The test temperatures were sometimes high, over 40 celsius. We believe some of these cells may have experienced internal temperatures hot enough to vaporize the electrolyte and "dry out" part of the cell stack. The subject test program was devised to resolve some of these inherent problems. A representative number of 2000-ampere-hour cells were activated with varying quantities of electrolyte for the purpose of optimizing electrolyte quantity in relation to overall cell performance at a predetermined constant temperature and rate of discharge. After electrolyte optimization was determined, additional 2000-ampere-hour cells were constructed and tested to characterize performance at various rates and temperatures. Abuse tests consisting of 100% cell reversal were also performed on the 2000-ampere-hour cells.

Occurring simultaneously with the 2000-ampere-hour characterization tests, 200-ampere-hour cells were constructed and tested in an effort to determine performance under the following conditions: 1) discharging at various rates, 2) intermittent discharging, 3) cyclic discharging, and 4) extended storage at open circuit followed by discharge at various rates.

A total of 26 calcium/thionyl chloride, low surface area cells were designed and fabricated for the purpose of testing as described above. These cells were made and tested according to the following schedules.

1. Six cells of 2000 ampere-hour capacity were activated with various quantities of electrolyte to determine the amount needed to achieve the highest specific energy.

GENERAL INTRODUCTION (continued)

2. Twelve cells of 2000 ampere-hour capacity were discharged, each at a different current or temperature to determine general performance characteristics.
3. Eight cells of 200 ampere-hour capacity were discharged intermittently to characterize discharge patterns over a longer time span.
4. Two cells of 2000 ampere-hour capacity were reverse discharged until they would no longer accept a current. This test was performed to demonstrate performance and safety under adverse conditions.

SECTION II

CELL COMPONENT DISCUSSION

1.0 INTRODUCTION

Two cell designs were used in these tests, a 2000 ampere-hour design for the short term discharges, and a 200 ampere-hour design for the longer duration tests.

Components are described in the following paragraphs.

2.0 CATHODES

The cathodes used in these cells are made from a mixture of 90% Shawinigan Acetylene Black and 10% TFE-30 Teflon on a current collector of foamed Nickel 200 with a sheet of 0.03 centimeter (.012 inch) thick Dexter non-woven glass separator molded on to the surface to prevent short circuits from the cut surface of the nickel foam.

The Acetylene Black and the Teflon were bound into a workable paste with an organic solvent. The paste was then worked into a porous current collector of 2.3 to 2.7 percent nickel and a layer of glass separator was applied to both sides. The assembly was then baked in an oven at 340 celsius.

The final carbon density of the cathodes ranged between 0.06 and 0.09 grams per cubic centimeter. The cathodes measured 0.64 x 16.5 x 31.8. However, the edges were flattened to 0.3 centimeter (1/8 inch) and framed with 0.01 centimeter (0.005 inch) thick nickel 200 foil. This left a useable cathode of 15 x 30 centimeters, 288 cubic centimeters (6 x 12 inches, 17.6 cubic inches). A cathode utilization of 0.30 ampere-hours per cubic centimeter (4.9 ampere-hours per cubic inch) would have yielded 1900 ampere hours from the 2000 ampere-hour cells, and 180 ampere-hours from the 200-ampere-hour cells. A cathode utilization of 0.40 ampere-

2.0 CATHODES (continued)

hours per cubic centimeter (6.6 ampere-hours per cubic inch), the best seen during previous tests, would have yielded 2500 ampere-hours from the 2000-ampere-hour cells, and 230 ampere-hours from the 200-ampere-hour cells. The weight of each cathode was 117 grams (0.258 pounds).

3.0 ANODES

The electrodes for both sizes of cells were identical, the difference in the cells had 23 anodes and the 200-ampere-hour cells had three anodes. Each anode was made by placing two pieces of calcium foil together (edge to edge) and joining them by spotwelding a nickel 200 strip across each end. The pieces of foil were 0.13 x 8.26 x 31.8 centimeters (0.050 x 3.25 x 12.5 inches). Due to the design of the cathode, the usable surface of the anode was cut to 15 x 30 centimeters (6 x 12 inches) to give a total usable volume of 59 cubic centimeters (3.6 cubic inches) of calcium per anode. The capacity was 122 ampere-hour per anode for a total of 2790 ampere-hours in the 2000 ampere-hour cells and 365 ampere-hours in the 200-ampere-hour cells. Each anode weighed 108 grams (0.238 pound).

The calcium for the anodes was purchased from PMF Alloys came in a work hardened state due to the process of rolling the calcium billets into a foil. The impurities of these 38 billets are, as a mean:

iron	-	85 ppm
manganese	-	146 ppm
nitrogen	-	1445 ppm
aluminum	-	1300 ppm
magnesium	-	2975 ppm

3.0 Anodes (Continued)

heating the foil to 460 Celsius (860 Fahrenheit). This yielded a very workable piece of calcium; however, due to the lack of humidity control at the contractor's facility, the foil was rather oxidized.

4.0 Separator

The electrodes were separated with 0.30 millimeter (0.012 inch) thick Dexter woven glass "U"-wrapped around each cathode. Each separator weighed 6.4 grams (0.014 pound).

5.0 Vent System

The vent mechanism consisted of a stainless steel body with a 2.5 centimeter (one inch) diameter nickel rupture disk sandwiched between the body and a retainer ring. The rupture disk and retainer ring were sealed to the body by heliarc welding the periphery of the body assembly. A vacuum support device was inserted between the rupture disk and the vent body during assembly. This assembly was connected to the cell by a standard stainless steel pipe, 0.6 centimeters (0.25 inches) diameter by 4 centimeters (1.5 inches) long. This assembly weighed 42 grams (0.093 pound). See Figure 1.

The vent mechanism was designed to open at approximately 10 atmospheres (150 pounds per square inch). The mechanism was not designed to reclose.

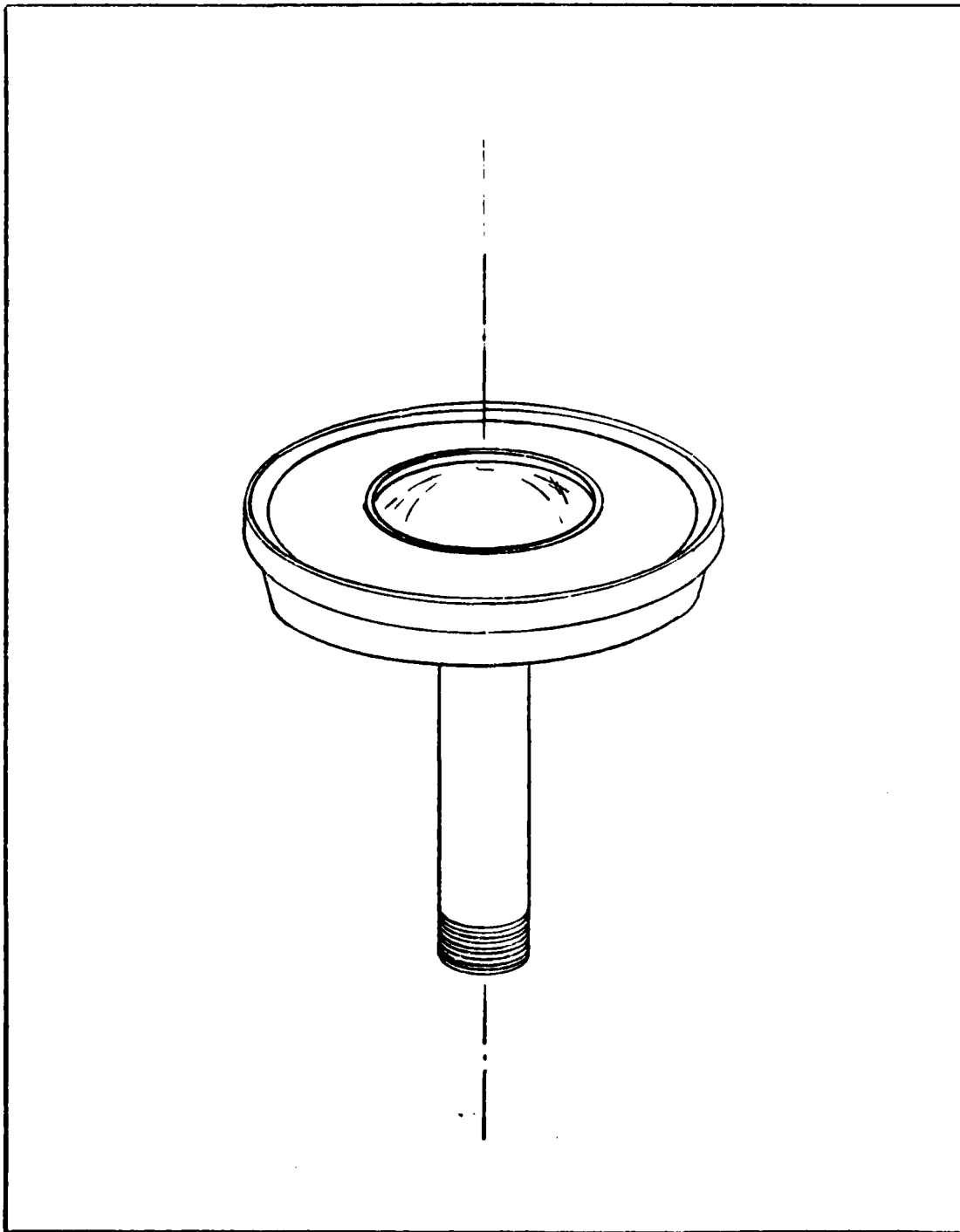


Figure 1. Vent Mechanism

6.0 Terminals

The terminals of the cells contained a feedthrough of Nickel-52, 4 millimeters (0.16 inches) diameter by 25 millimeters (1.0 inch) long, insulated from the cell cover by Corning 9010 glass.

7.0 Containers

The 2000-ampere-hour cells were housed in a container 17 x 19 x 35 centimeters (6.7 x 7.4 x 13.8 inches) with 0.46 millimeter (0.018 inch) thick walls. The 200-ampere-hour cell container was 2.1 x 17 x 35 centimeters (0.82 x 6.7 x 13.8 inches) with 0.46 millimeter (0.018 inch) thick walls.

The original cell cases for the 2000-ampere-hour cells weighed 1.23 kilograms (2.71 pounds) but were not able to tolerate the vacuum needed to activate the cell. Only cells numbered 2001 through 2006 used this case. The remaining cells were built using containers with stainless steel channel shaped reinforcement ribs welded onto the surface. This increased the tolerance of the container to vacuum but also increased the weight of the cell to 2.85 kilograms (6.28 pounds). The volume of the cell is 11.3 cubic decimeters (691 cubic inches).

The 200-ampere-hour cell case weighed 0.56 kilograms (1.23 pounds) and had a volume of 1.25 cubic decimeters (76 cubic inches).

8.0 Insulators

The cell stack was insulated from the cell case with a layer of 0.25 millimeter (0.010 inch) thick Teflon TFE, doubled on the edges. Teflon, 0.8 millimeter (0.03 inch) thick, was used to separate the terminal strips above the electrodes. The Teflon weighed 205 grams (0.452 pounds) in the 2000-ampere-hour cells and 145 grams (0.320 pounds) in the 200-ampere-hour cells.

SECTION III

CELL DESIGN

1.0 Introduction

The 2000-ampere-hour cell design was used in the short-term discharge tests and the 200-ampere-hour design was used for the tests of longer duration.

2.0 2000-Ampere-Hour Design

These cells were made using 22 cathodes and 23 anodes and housed in the cell case described earlier and pictured in Figure 2. A lead of 0.13 x 6.4 millimeters (0.005 x 0.25 inch) Nickel-200 was spotwelded to the nickel border along the top of each electrode. Teflon insulators were used to separate the leads coming off the cathodes, the leads passing through slits in the Teflon. The leads were divided into two groups of eleven each and spotwelded together. Three similar leads were used to connect each of these two groups to the negative terminal of the cell. A similar procedure was used on the anodes. A reference electrode of calcium foil was wrapped in separator material and inserted between an outside anode and the cell case insulator. A Teflon insulator shielded the terminal leads from the cell cover.

The cover was heliarc welded to the case and then fitted with a three-way valve, a vent, and a pressure transducer with a range of 0 to 7 atmospheres (0 to 100 pounds per square inch).

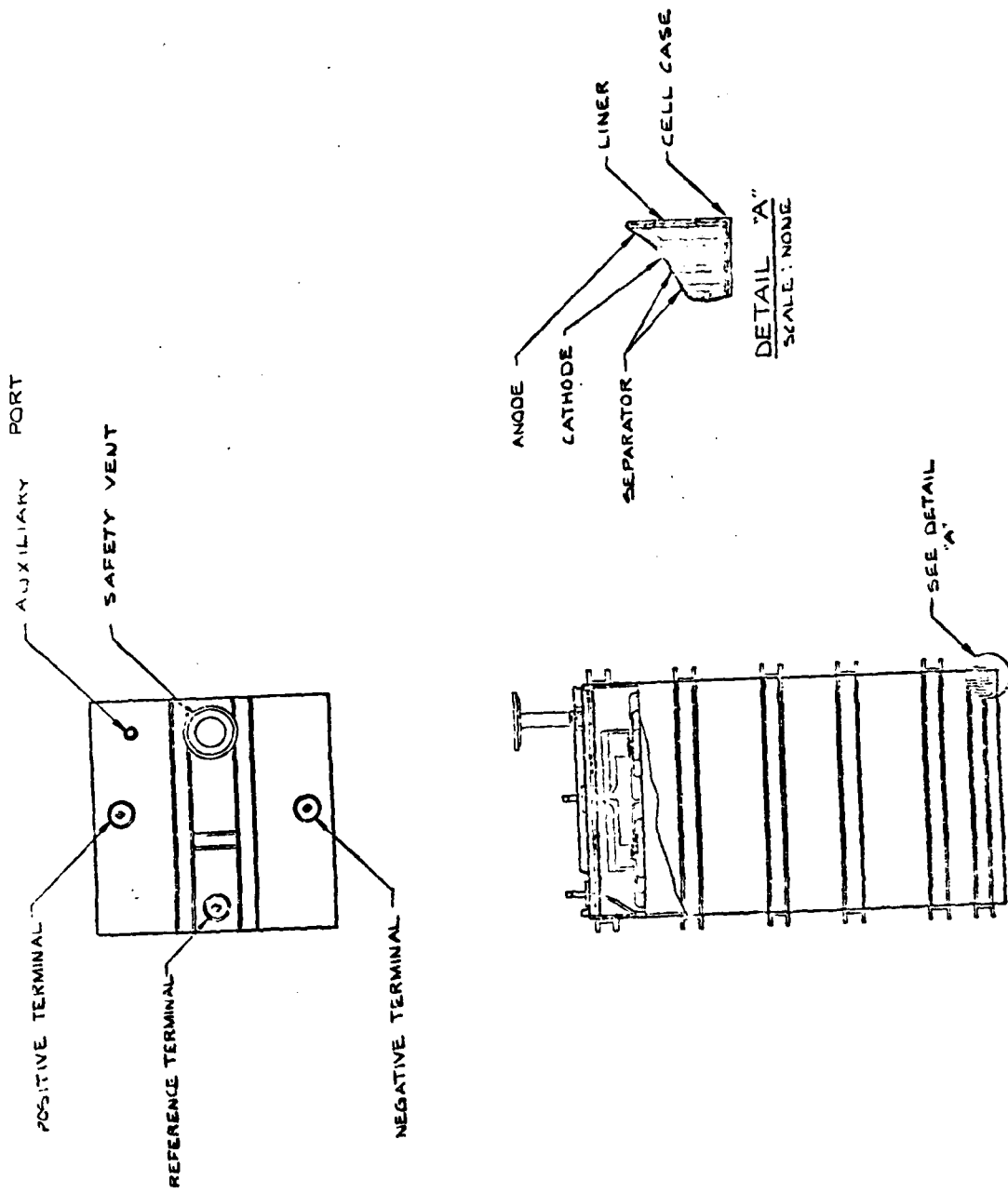


Figure 2. 2000-Ampere-Hour Cell

2.0 2000-Ampere-Hour Design (Continued)

The cells were then bound in a 1.3 centimeter (0.5 inch) thick aluminum restraining fixture to prevent the possibility of high pressures from forcing the cell walls out beyond the required dimensions and allowing the cell stack to shift.

3.0 200-Ampere-Hour Design

The 200-ampere-hour cells contained only two cathodes and three anodes. Assuming a cathode-limited system, the capacities of these cells should have been nine percent of the capacities of the larger cells. These cells were built similarly to the 2000-ampere-hour cells with the exception of the number of anodes and cathodes as described above. See Figure 3.

Since only two (2) $\frac{1}{2}$ -inch NPT ports were available on these cells, the vent was connected to the three-way valve rather than directly to the cell cover.

4.0 Discussion

The 2000-ampere-hour cell design had an effective cathode volume of 6490 cubic centimeters (396 cubic inches) and a surface area of 20,400 square centimeters (3170 square inches). The anode volume was 1360 cubic centimeters (82.8 cubic inches).

The 200-ampere-hour cell design had an effective cathode volume of 590 cubic centimeters (36 cubic inches) and an anode volume of 177 cubic centimeters (10.8 cubic inches). The surface area was 1860 square centimeters (280 square inches).

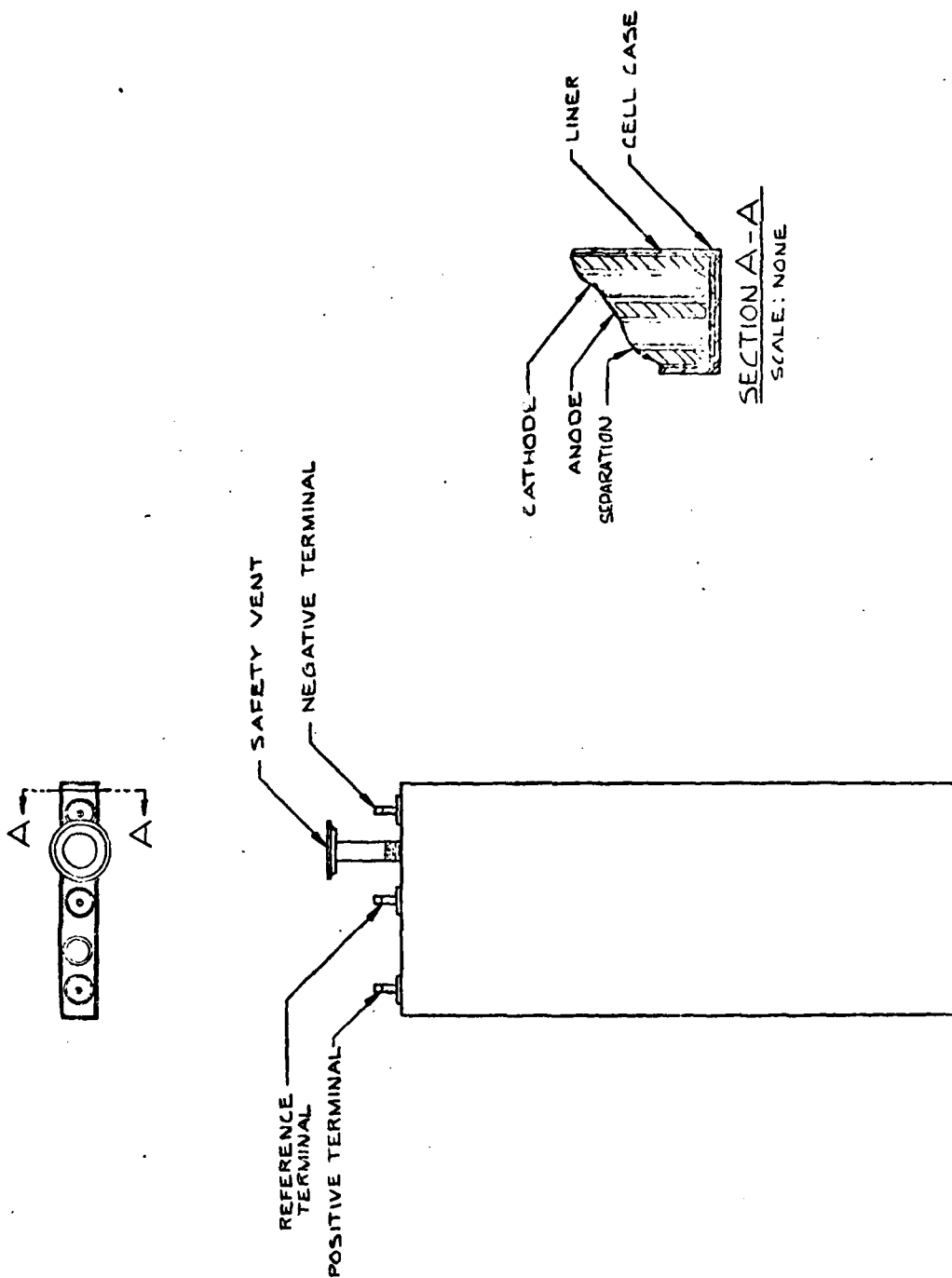


Figure 3. 200-Ampere-Hour Cell

4.0 Discussion (Continued)

At 2.04 ampere-hours per cubic centimeter (33.4 ampere-hour per cubic inch), the anode capacities were 2770 ampere-hours from the 2000 ampere-hour cells and 361 ampere-hours from the 200-ampere-hour design.

At 0.30 ampere-hours per cubic centimeter (4.9 ampere-hours per cubic inch) the cathode capacities are 1900 ampere-hours from the 2000-ampere-hour design and 180 ampere-hours from the 200-ampere-hour design.

With a cathode efficiency of 0.40 ampere-hours per cubic centimeter (6.6 ampere-hours per cubic inch), the capacities are 2600 ampere-hours from the 2000-ampere-hour design and 236 ampere-hours from the 200-ampere-hour design.

The dry 2000-ampere-hour cells weighed 6.6 kilograms (14.5 pounds) without support ribs and 8.1 kilograms (17.9 pounds) with ribs. The dry 200-ampere hour cells weighed 1.175 kilograms (2.59 pounds). The cell volumes were 11.3 cubic decimeters (69 cubic inches) for the 2000 ampere-hour cells and 1.25 cubic decimeters (76 cubic inches) for the 200-ampere-hour design.

SECTION IV

ACTIVATION

1.0 Electrolyte

The electrolyte used in these cells was 1.5 Formal lithium tetra chloroaluminate in thionyl chloride with five percent sulfur dioxide. The electrolyte was packaged in 20-liter (five-gallon) glass carboys. The electrolyte was transferred from the carboys to individual containers used for activating the cells in an argon atmosphere.

2.0 Apparatus

Each cell was activated by evacuating the cell through a three-way valve, and then turning the valve to use atmospheric pressure to force the electrolyte from a glass container through a Teflon tube connecting the valve to the bottom of the glass container. The pressure in the glass electrolyte container was equalized with argon during activation.

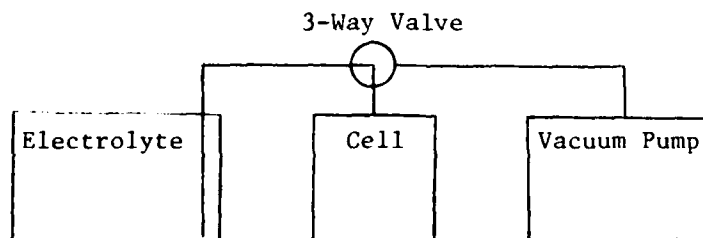


Figure 4. Activation Schematic

SECTION V

CELL COMPONENT STOICHIOMETRY AND
ELECTROLYTE QUANTITY OPTIMIZATION

1.0 INTRODUCTION

It should be noted that due to the rather limited history of this system, there are several uncertainties in the design. The cathode matrix utilization has characteristically varied in the past, due in part to electrolyte type and cell temperature. Reference Section I. Such variation obviously made cell balance problematic. Electrodes used in these cells were balanced for cathode utilization of 0.40 ampere-hours per cubic centimeter (6.6 ampere-hour per cubic inch), equivalent to a cell capacity of 2600 ampere-hours. The anodes were given seven percent excess.

The basis for assuming the above mentioned cathode utilization is primarily an improved control over the parameters assumed responsible:

1. The cell temperature was to be controlled.
2. The electrolyte was prepared with the same composition and by the same vendor as that which produced the 0.4 ampere-hour per cubic centimeter utilization in previous tests.
3. The apparent current density was lower than previous tests.

Calculations showed a volume of electrolyte equal to 6.5 liters (1.7 gallons) was needed to fill the voids in the cathodes and separator; i.e., to wet the cell stack. Further calculations indicated a volume of

1.0 INTRODUCTION (continued)

9.0 liters (2.4 gallons) would completely fill the cell. In order to eliminate the danger of cell case rupture due to hydraulic pressure, 8.0 liters (2.1 gallons) was determined to be a safe maximum electrolyte volume.

2.0 2000-Ampere-Hour Cell Tests

Six cells of 2000-ampere-hour design (Cell numbers 2001 through 2006) were used to determine the optimum electrolyte volume. Two cells were to receive 8.00 liters (2.11 gallons) of electrolyte, two were to receive 7.25 liters (1.92 gallons), and two were to receive 6.50 liters (1.72 gallons). The first six 2000-ampere-hour cell cases did not incorporate external supports; therefore, the vacuum pulled was requirably less. Consequently, some of the cells did not receive the volume of electrolyte intended and will be noted accordingly. See Figure 5.

After activation the cells were allowed to stand at open circuit a day. The cells were then discharged through a constant resistive load. The load was applied and removed by an automated discharge controller. The cells were discharged in 20 Celsius water baths and data was taken concerning cell potential, current, cell surface temperature, water bath temperature, and ambient temperature.

2.1 Cell Number 2001

Cell Number 2001 received 6.8 liters (1.8 gallons) of electrolyte. After a day of discharging the load was adjusted to 5.2 amperes. A temporary power failure interrupted the discharge after one week as indicated in Figure 6. The average water bath temperature was 18 Celsius (64 Fahrenheit) and the average current

RESULTS OF ELECTROLYTE QUANTITY TESTS

CELL NUMBER	ELECTROLYTE VOLUME L.(GAL)	DIS. CUR AMPERES	WATER BATH TEMPERATURE CELSIUS(°F)	* DISCHARGE TIME CAPACITY HOURS AMP-HOURS	* ENERGY WATT-HOURS	* EMER. WH/DMF3	DENS.* WH/INT3	SPEC.* WH/KG.	ENER.* WH/LB.	CUT OFF POTENTIAL VOLTS
2001	6.8(1.8)	4.70	18 (64)	190 321 392	2388 4116 4858	211 364 430	3.46 5.97 7.05	134 231 273	61 105 124	2.70 2.50 2.00
2002	5.5(1.7)	5.76	26 (79)	202 244 287	3464 4066 4542	307 360 402	5.03 5.90 6.59	200 235 263	91 107 119	2.70 2.50 2.00
2003	7.9(2.1)	4.92	17 (63)	203 367 429	2076 4995 5648	255 442 500	4.18 7.24 8.20	147 255 288	67 116 131	2.70 2.50 2.00
2004	6.7(1.8)	4.75	18 (64)	240 348 419	3440 4839 5336	304 411 472	4.93 6.74 7.74	194 262 301	88 119 137	2.70 2.50 2.00
2005	7.3(1.9)	4.72	17 (63)	126 345 416	2135 4343 5028	189 402 463	3.10 6.59 7.59	115 244 281	52 111 127	2.70 2.50 2.00
2006	6.3(1.7)	6.02	25 (77)	191 220 275	3341 3934 4482	296 346 397	4.85 5.70 6.51	197 231 264	89 105 120	2.70 2.50 2.00

Figure 5

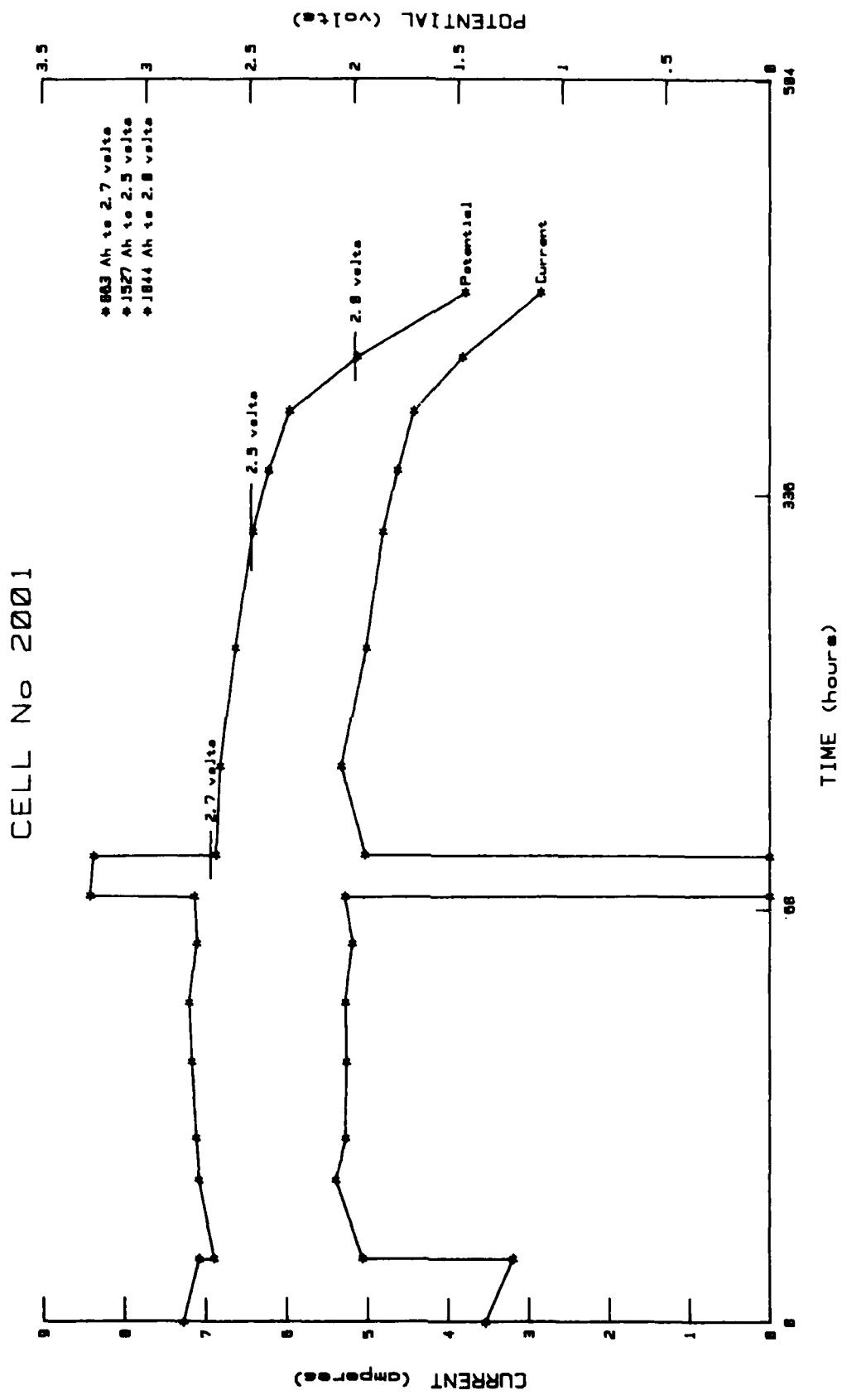


Figure 6.

2.1 Cell Number 2001 (continued)

was 4.70 amperes. After 392 hours the cell potential fell below 2.0 volts. For further information, see Figures 5 and 6.

2.2 Cell Number 2002

Cell Number 2002 received 6.5 liters (1.7 gallons) of electrolyte. The initial current was higher than intended, probably due to the higher temperature of the water bath. See Figure 5. The capacity yielded from this cell was lower than expected, probably due to the low electrolyte quantity. See Figure 7.

2.3 Cell Number 2003

After a day of discharging the load on cell number 2003 was adjusted to 5.2 amperes. This cell contained the largest quantity of electrolyte (7.9 liters, 2.1 gallons) as shown in Figure 5. It also yielded the highest capacity. See Figure 8.

2.4 Cell Number 2004

Cell Number 2004 received 6.7 liters (1.8 gallons) of electrolyte. After 32 hours of discharge, the cell potential and current inexplicably began to decay (see Figure 9). This decay continued until approximately 48 hours at which time full recovery began. We cannot completely explain the cause of the problem; however, it was probably a partial internal short which occurred spontaneously and later opened.

Due to the complete and quick recovery of the cell potential above 2.7 volts, the voltage cut-off requirement of 2.7 volts

CELL No 2002

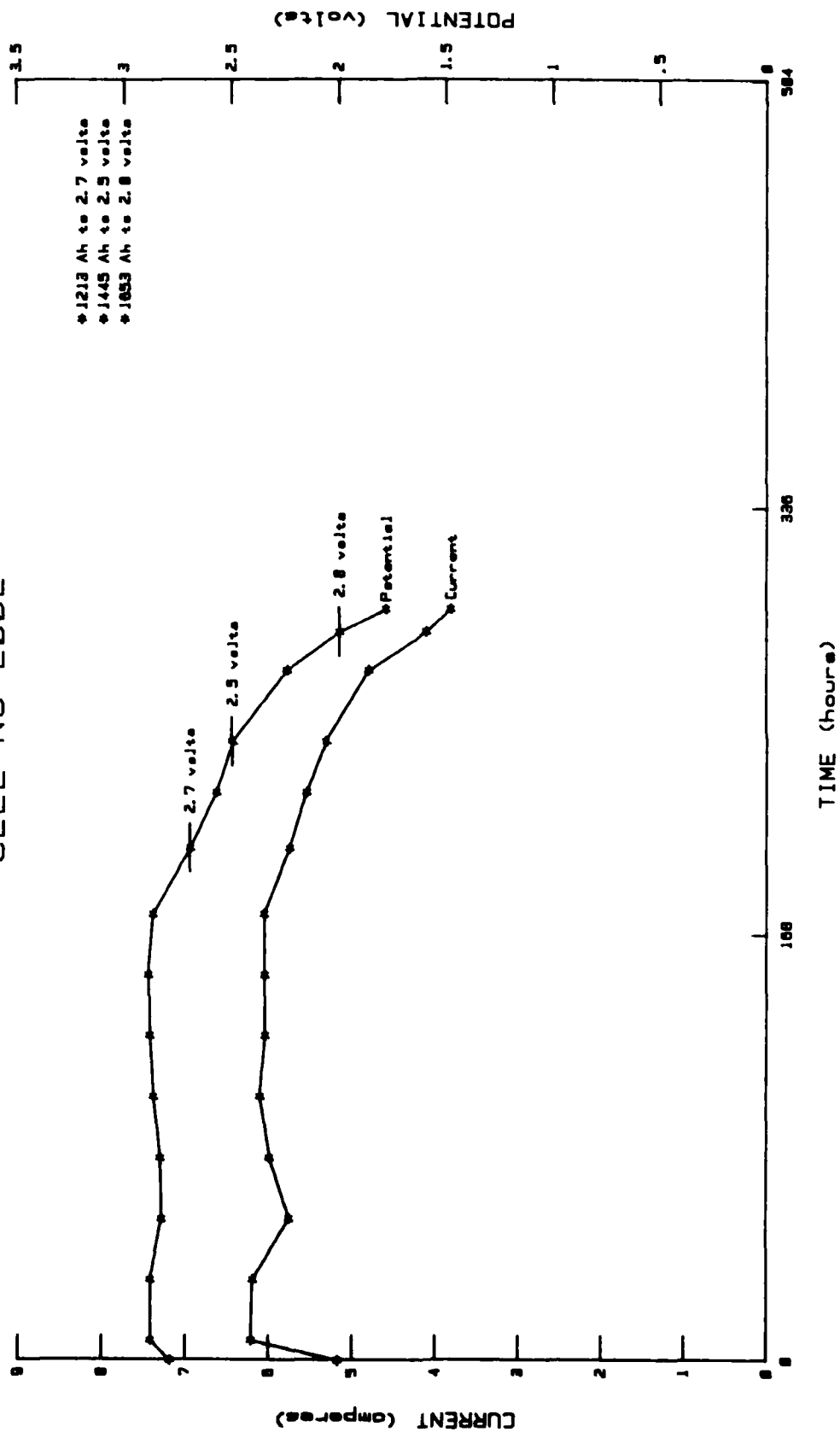


Figure 7

CELL No 2003

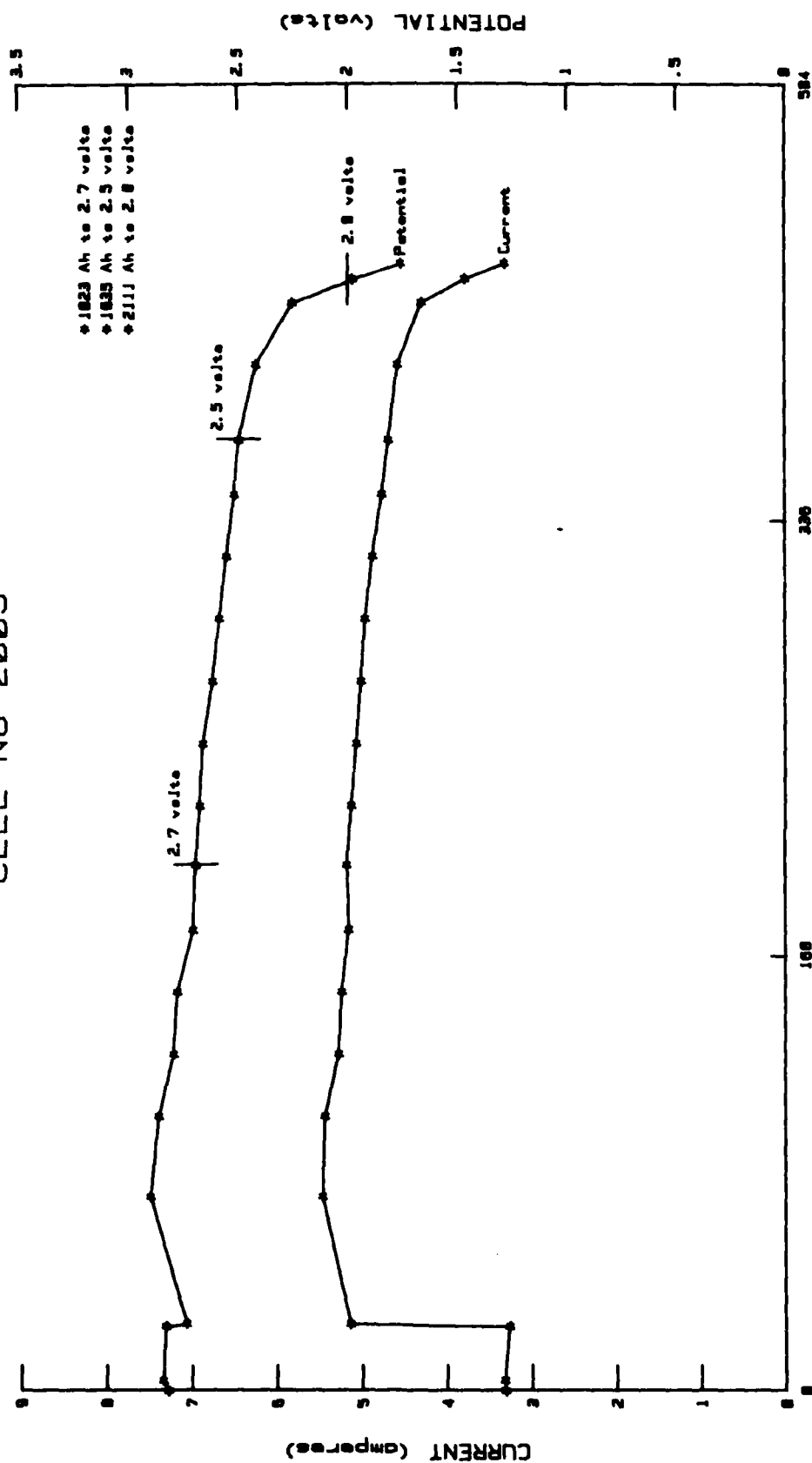
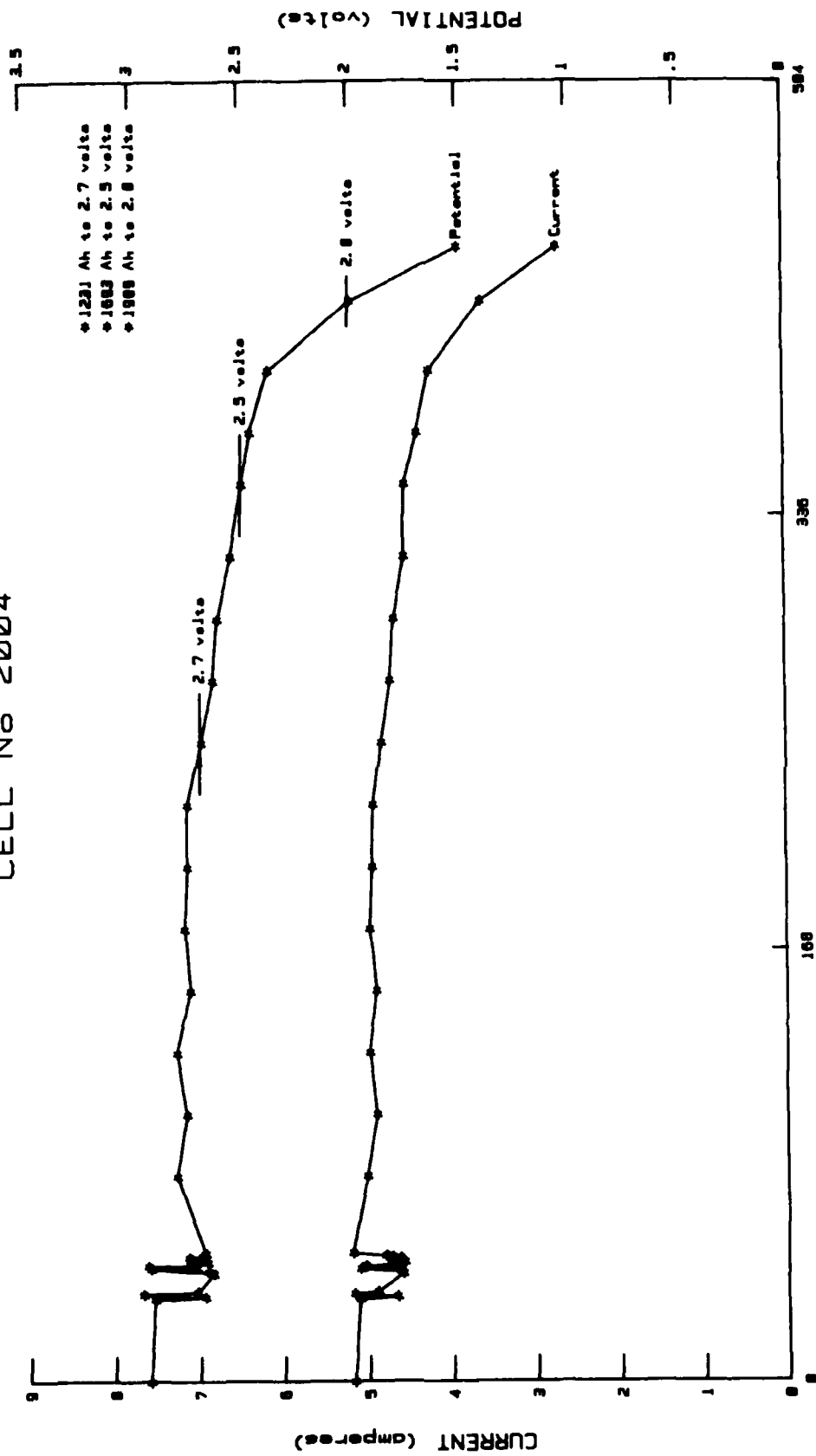


Figure 8

CELL No 2004



TIME (hours)
Figure 9

2.4 Cell Number 2004 (continued)

was not considered reached at this time (see Figure 9). The cell yielded the highest specific energy of the electrolyte optimization test cells.

2.5 Cell Number 2005

The cell was discharged at an average current of 4.72 amperes to a potential of 1.5 volts. The cell received 7.3 liters (1.9 gallons) of electrolyte and the average water temperature was 17 Celsius (63 Fahrenheit). See Figures 5 and 10 for additional information on this cell.

2.6 Cell Number 2006

The initial current for this cell was higher than any of those previously mentioned. Its water bath temperature was also high. This cell received the least amount of electrolyte of all the electrolyte test cells, and the capacity was correspondingly low. See Figures 5 and 11 for additional information. After 51 hours of discharge, the cell potential fell to 2.596 volts. The cell potential recovered to 2.7 volts after 29 hours and later peaked at 2.866 volts. Due to a thermocouple failure, the water temperature during this time is not known, however, we feel it may have cooled enough to cause the problem. The capacity given to 2.7 volts is for the period ending at 191 hours into the discharge when the potential fell below 2.7 volts for the second time.

CELL No 2005

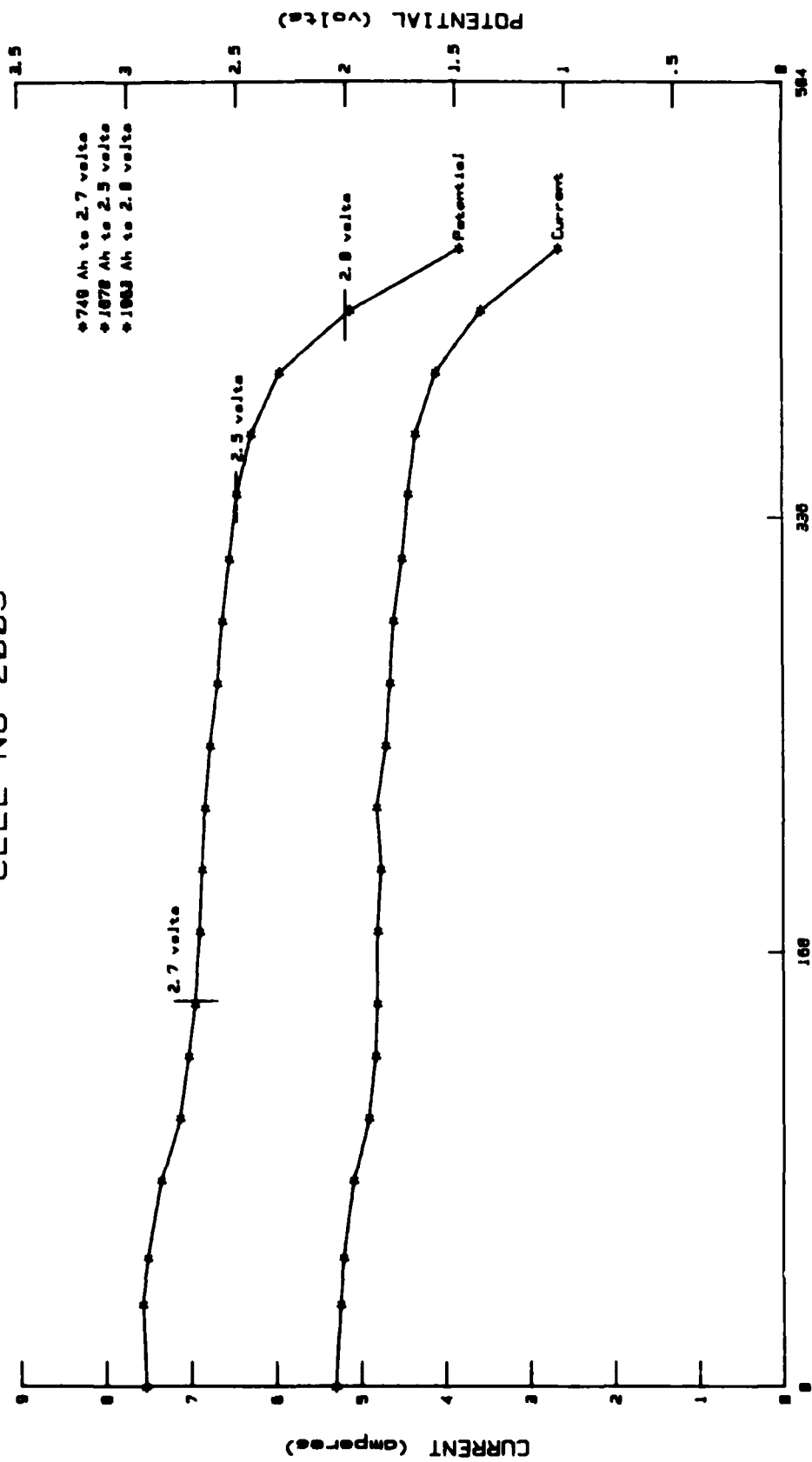


Figure 10

CELL No 2006

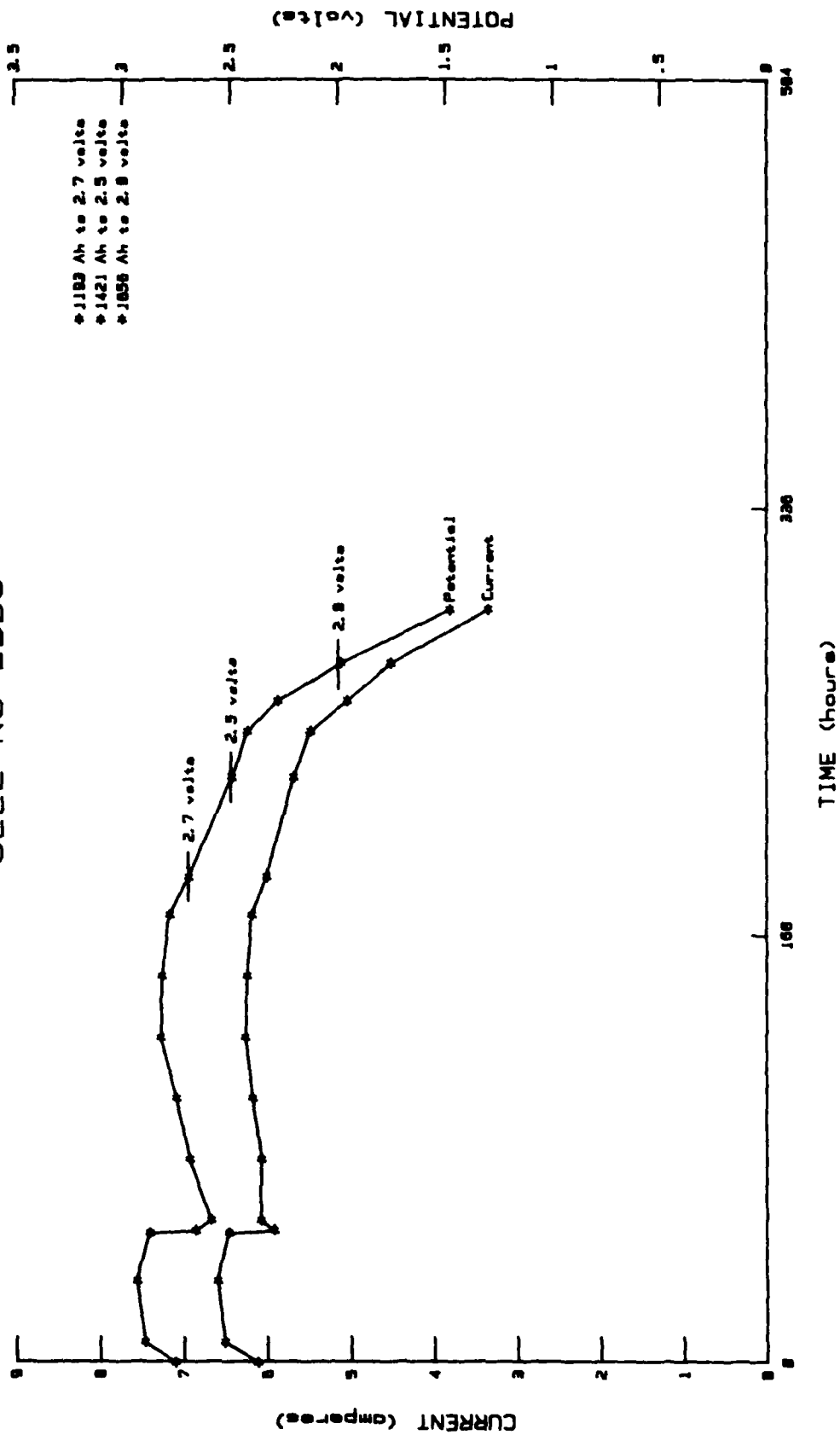


Figure 11

3.0 Conclusions

Cell number 2002 with 6.5 liters (1.7 gallons) of electrolyte yielded the highest energy density above 2.7 volts. Cell number 2003 with 7.9 liters (2.1 gallons) of electrolyte showed the highest energy density to 2.5 volts and 2.0 volts.

Cell number 2002 also had the highest specific energy above 2.7 volts. When the cut off potential was lowered to 2.5 volts and to 2.0 volts, cell number 2004 with 6.7 liters (1.8 gallons) of electrolyte performed the best.

The optimum electrolyte volume involved a compromise between the highest energy density and the highest specific energy. With a 2.7 volt cut off, an electrolyte volume of 6.5 liters (1.7 gallons) was evidently best. However, with lower cut-off potentials, a compromise between the 6.7 liter volume producing the highest specific energy and the 7.9 liter volume producing the highest energy density is needed. An electrolyte volume of 7.0 liters (1.8 gallons) was chosen as optimum for the remaining characterization tests of 2000-ampere-hour cells. See Figures 12 and 13.

ELECTROLYTE VOLUME OPTIMIZATION

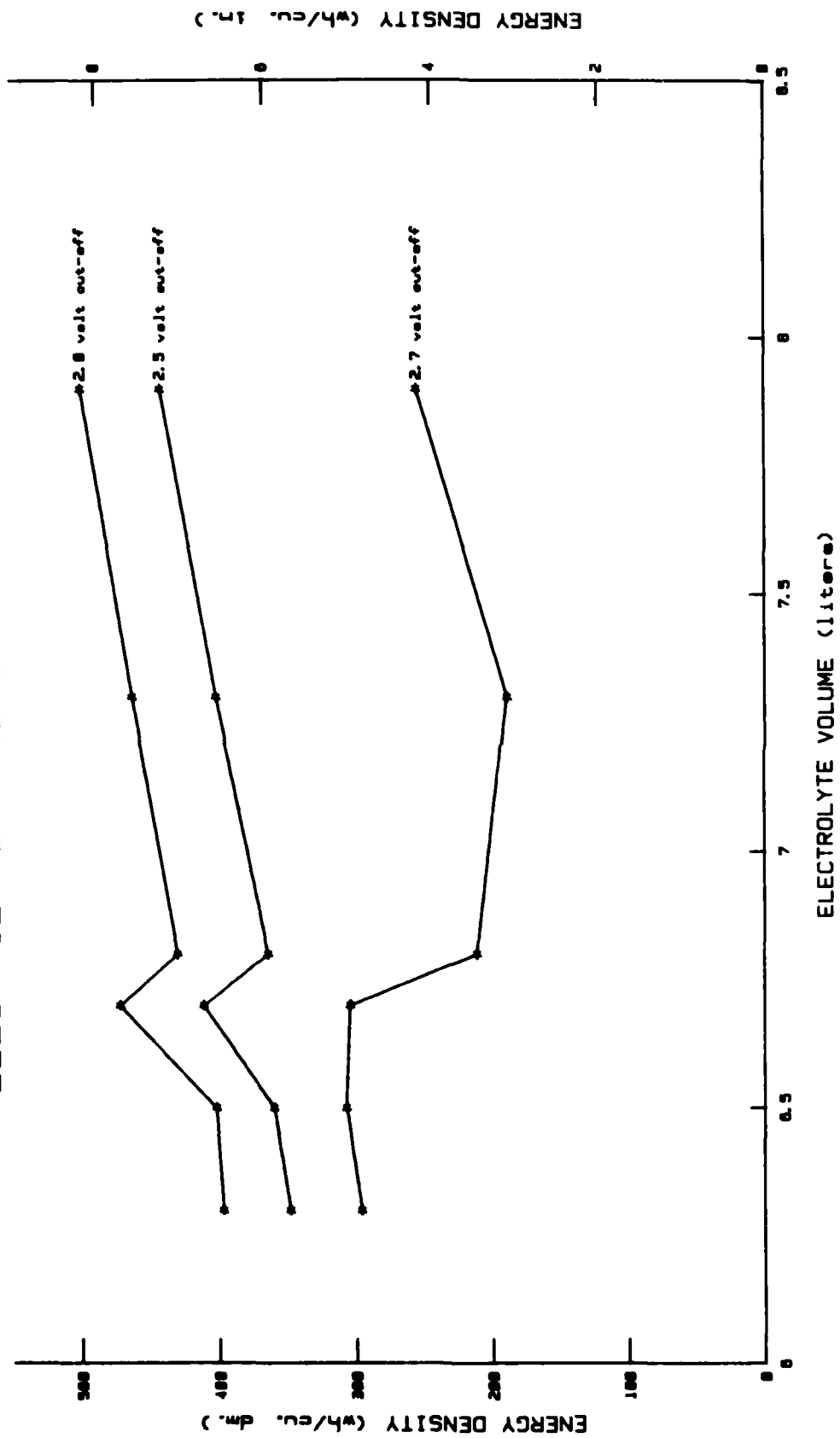


Figure 12.

ELECTROLYTE VOLUME OPTIMIZATION

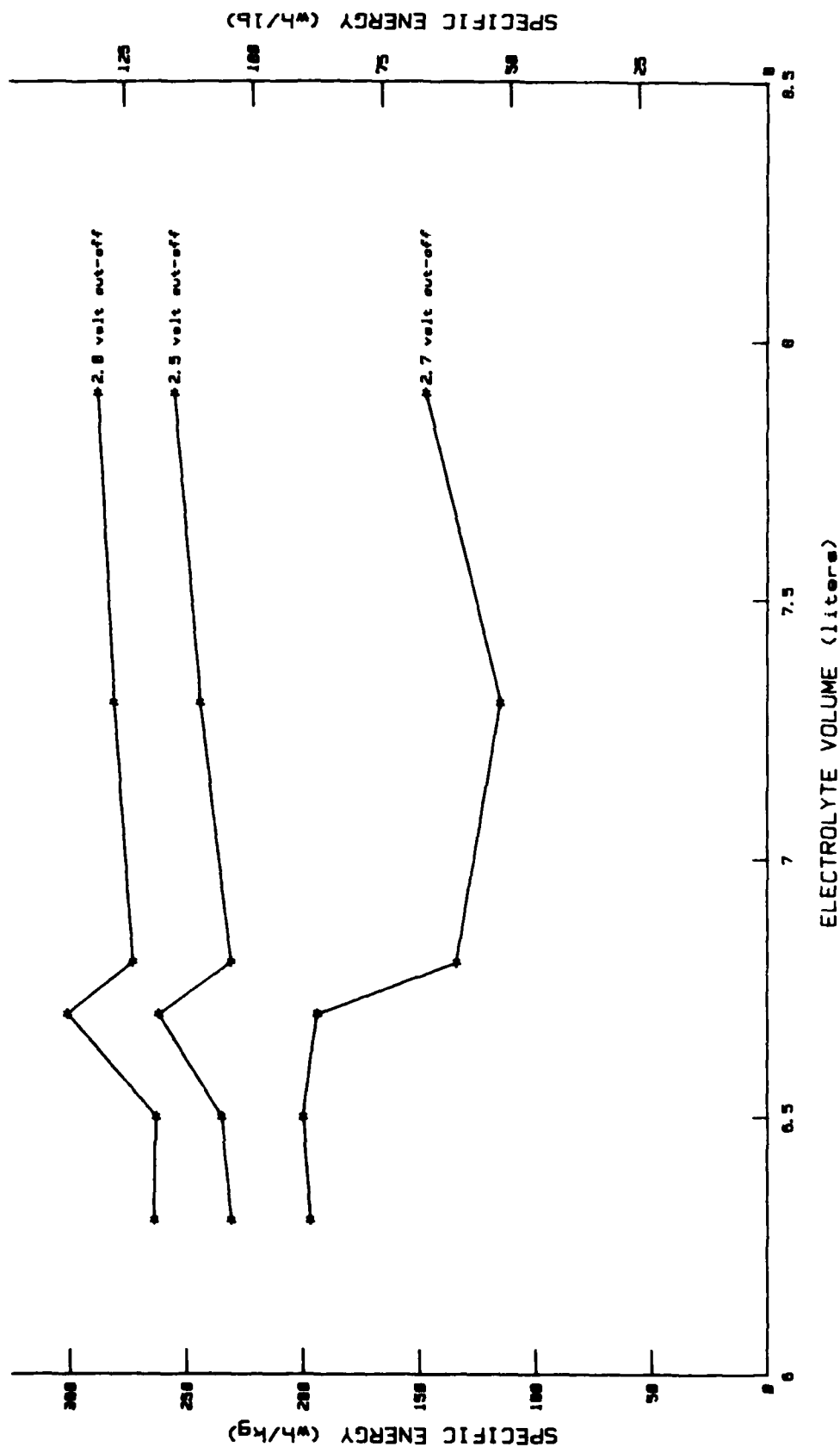


Figure 13

SECTION VI
2000 AMPERE-HOUR CELL CHARACTERIZATION

1.0 INTRODUCTION

Twelve cells were built and tested to determine performance characteristics. These were divided into two groups for testing.

1. Eight cells were discharged at four different rates, two cells at each rate. The test temperature of these cells was constant.
2. Four cells were discharged at a constant rate and two different temperatures; two at each temperature.

The cells were discharged using the automatic discharge controller mentioned in Section V, Paragraph 2.0 on Page 16. We experienced problems with this discharge controller during the discharges of cells 2009, 2010, 2011, and 2012. After futile attempts to correct the problems we completely bypassed the controller and wired a resistor directly to the cells. None of the cells discharged under phase 2 of this Section used the discharge controller.

The temperatures of the cells were controlled by the use of water baths controlled by either a refrigeration unit or heater elements. Data was taken concerning cell potential, current, cell surface temperature, water bath temperature, and ambient temperature.

Prior to activation, the cells were placed in a water bath environment. Cell temperature was monitored on the outside of the cell and approximately one inch above the water level. A more ideal point for monitoring cell

1.0 INTRODUCTION (continued)

temperature would have been on the side, near the middle of the cell; however, it would not have been possible to properly insulate the thermocouple from the direct water temperature influence with the mid-cell approach.

2.0 TESTS AT VARIOUS DISCHARGE RATES

The eight cells for this test were to be discharged according to the following schedule.

- 1) Cell Numbers 2009 and 2010 were to be discharged in 123 hours at 16.25 amperes.
- 2) Cell Numbers 2011 and 2012 were to be discharged in 192 hours at 10.4 amperes.
- 3) Cell Numbers 2014 and 2015 were to be discharged in 385 hours at 5.2 amperes.
- 4) Cell Numbers 2007 and 2009 were to be discharged in 769 hours at 2.6 amperes.

The water bath temperatures were maintained at a nominal 16 to 22 Celsius.

2.1 123 Hour Rate

While the average currents of these cells were only 9.0 amperes, the currents during the latter portion of the discharges were even higher than the desired 16 amperes. See Figures 14, 15, and 16.

RESULTS OF
2000-AMPERE-HOUR CELL CHARACTERIZATION

CELL NUMBER	ELECTROLYTE VOLUME L. (GAL)	DIS. CUR AMPERES	WATER BATH TEMPERATURE CELSIUS (F)	* DISCHARGE TIME HOURS	* CAPACITY AMP-HOURS	* ENERGY WATT- HOURS	* ENER. DENS.* WH/ DM ³	* SPEC. ENER.* WH/ IN ³	* WH/ KG.	* WH/ LB.	* CUT-OFF POTENTIAL VOLTS
2007	6.6(1.7)	2.10	22 (72)	662	1416	4159	368	6.03	238	108	2.70
				732	1561	4536	401	6.57	259	117	2.50
				774	1637	4714	417	6.83	269	122	2.00
2008	6.8(1.8)	2.19	19 (66)	684	1532	4445	393	6.44	250	113	2.70
				749	1665	4792	424	6.95	269	122	2.50
				776	1712	4901	434	7.11	275	125	2.00
2009	6.9(1.8)	9.00	18 (64)	37	240	674	60	.98	37	17	2.70
				144	929	2532	224	3.67	141	64	2.50
				181	1586	4048	358	5.07	225	102	2.00
2010	7.0(1.8)	9.01	16 (61)	12	80	222	20	.33	12	5	2.70
				145	940	2576	228	3.74	142	64	2.50
				185	1646	4221	374	6.13	232	105	2.00
2011	5.1(1.3)	7.45	20 (68)	18	87	253	22	.36	17	8	2.70
				42	202	569	50	.82	38	17	2.50
				139	1035	2781	246	4.03	185	84	2.00
2012	7.0(1.8)	7.76	22 (72)	42	197	578	51	.84	32	15	2.70
				155	1181	3268	289	4.74	181	82	2.50
				169	1311	3580	317	5.20	198	90	2.00
2013	7.0(1.8)	4.54	8 (46)	54	265	748	66	1.08	41	19	2.70
				322	1509	4058	359	5.88	224	102	2.50
				421	1911	4971	440	7.21	275	125	2.00
2014	7.0(1.8)	5.13	19 (66)	231	1232	3490	309	5.06	193	88	2.70
				322	1678	4642	411	6.74	256	116	2.50
				358	1837	4999	442	7.24	276	125	2.00
2015	7.0(1.8)	5.09	18 (64)	186	984	2784	246	4.03	154	70	2.70
				310	1604	4422	391	6.41	244	111	2.50
				340	1731	4691	415	6.80	259	117	2.00
2016	7.0(1.8)	4.79	8 (46)	42	222	626	55	.90	35	16	2.70
				297	1463	3864	342	5.61	213	97	2.50
				393	1884	4832	428	7.01	267	121	2.00
2017	7.0(1.8)	5.25	35 (95)	233	1236	3566	316	5.18	197	89	2.70
				244	1287	3706	328	5.38	205	93	2.50
				253	1327	3787	335	5.49	209	95	2.00
2018	7.0(1.8)	5.46	40 (104)	226	1252	3674	325	5.33	203	92	2.70
				233	1290	3777	334	5.47	209	95	2.50
				249	1359	3927	347	5.69	217	98	2.00

Figure 14

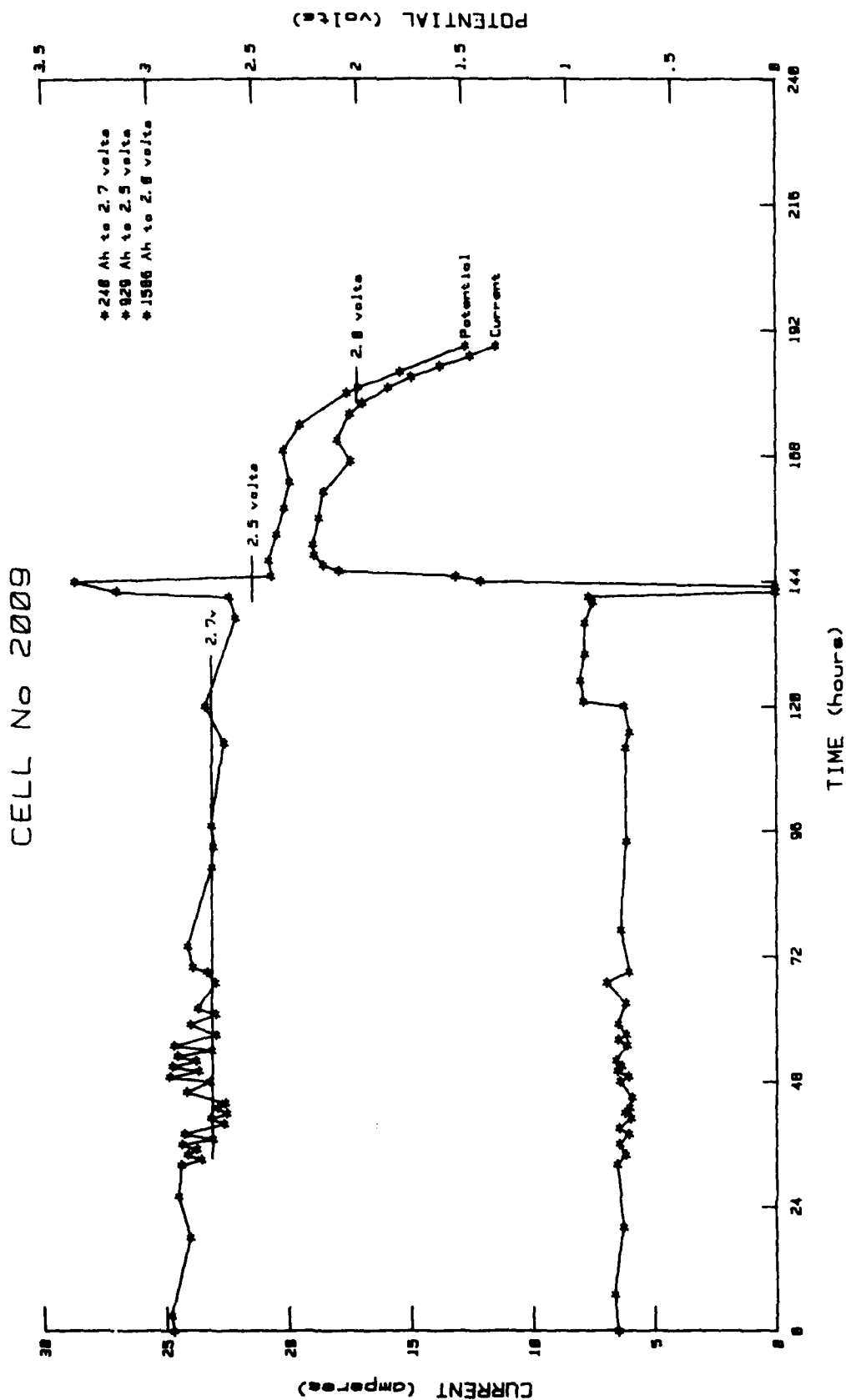


Figure 15

CELL No 2010

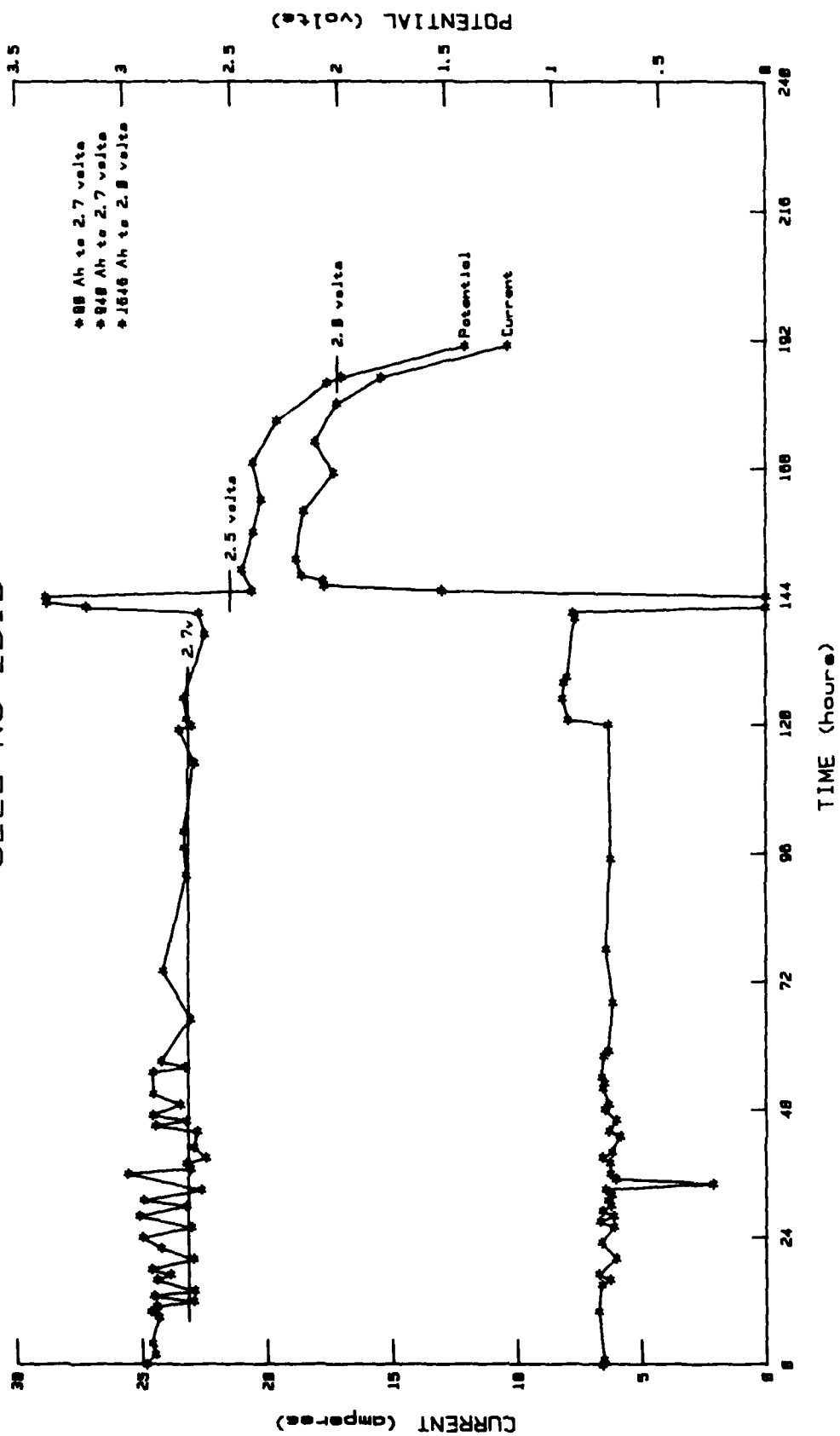


Figure 16

2.2 192 Hour Rate

The cell case for number 2011 leaked and admitted water to the cell when it was evacuated prior to activation. It received only 5.1 liters (1.3 gallons) of electrolyte. Cell number 2012 received 7.0 liters of electrolyte.

While the average currents of these cells were 7.4 to 7.8 amperes, the currents after the discharge controller was bypassed averaged 10.3 to 10.8 amperes. See Figures 14, 17 and 18.

2.3 385 Hour Rate

Cells numbered 2014 and 2015 were discharged at a nominal 5.1 amperes. Number 2014 lasted 358 hours and 2015 lasted 340 hours. See Figures 14, 19 and 20.

2.4 769 Hour Rate

The cells numbered 2007 and 2008 received 6.6 to 6.8 liters (1.7 - 1.8 gallons) of electrolyte. The discharge currents averaged 2.1 to 2.2 amperes and they only produced 1637 and 1712 ampere-hours of capacity. We believe this lower capacity was due to the lower electrolyte quantity. See Figures 14, 21 and 22.

3.0 TESTS AT VARIOUS TEMPERATURES

Four cells were discharged through resistors an average rate of 4.5 to 5.5 amperes:

- 1) Cells numbered 2013 and 2016 were to be discharged at 5 Celsius (40 Fahrenheit).
- 2) Cells numbered 2017 and 2018 were to be discharged at 40 Celsius (105 Fahrenheit).

CELL No 2011

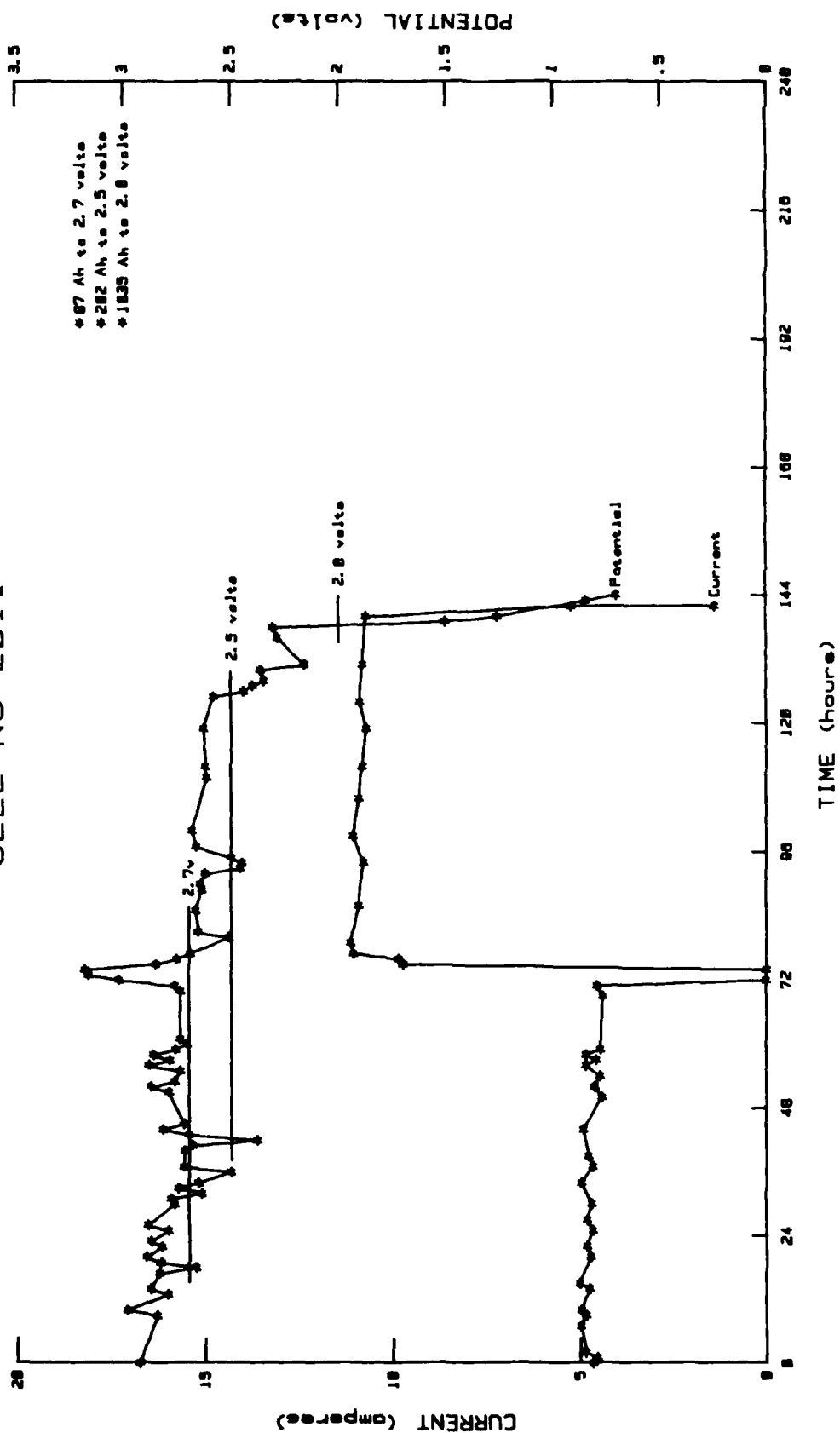


Figure 17

CELL No 2012

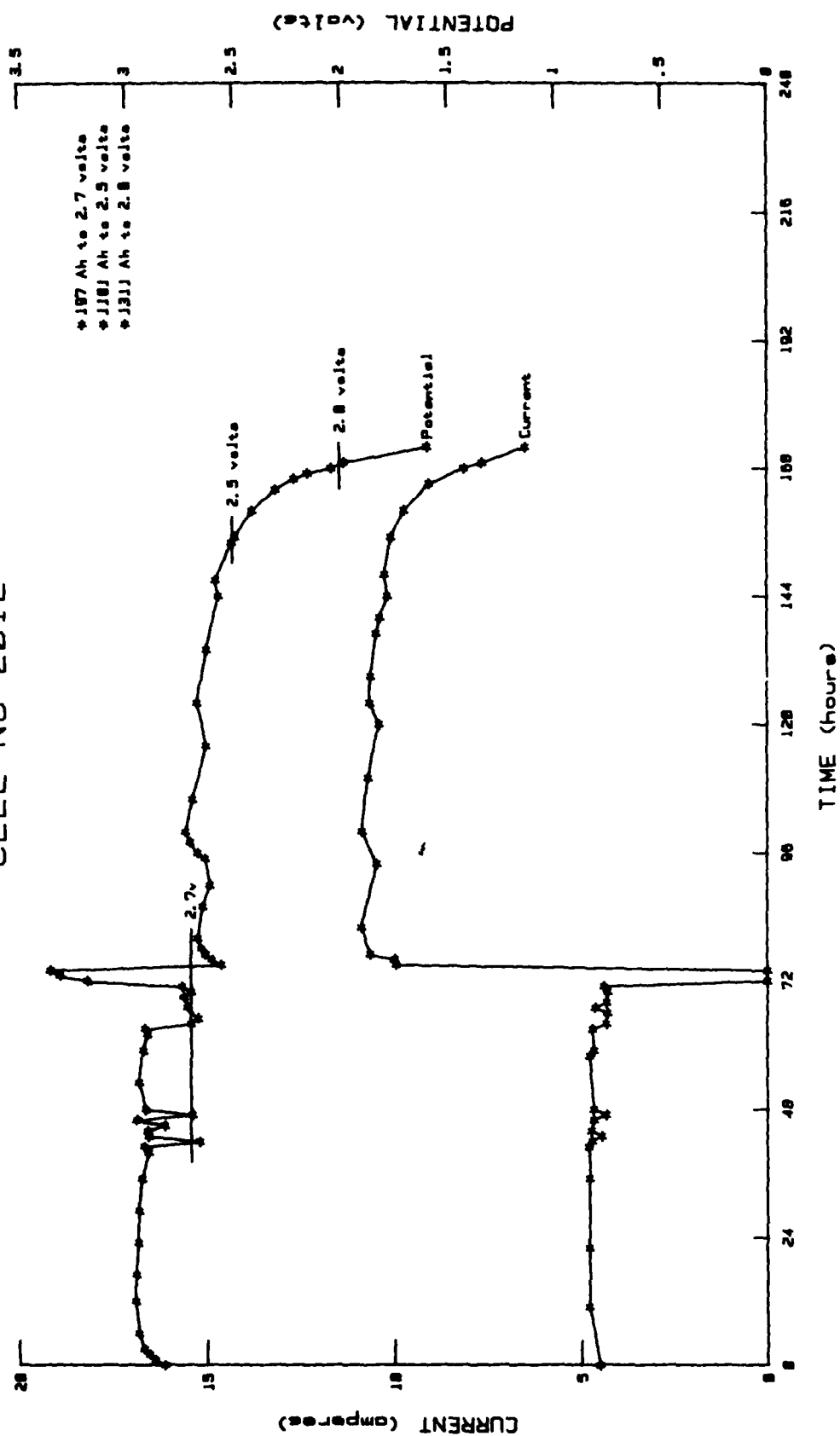
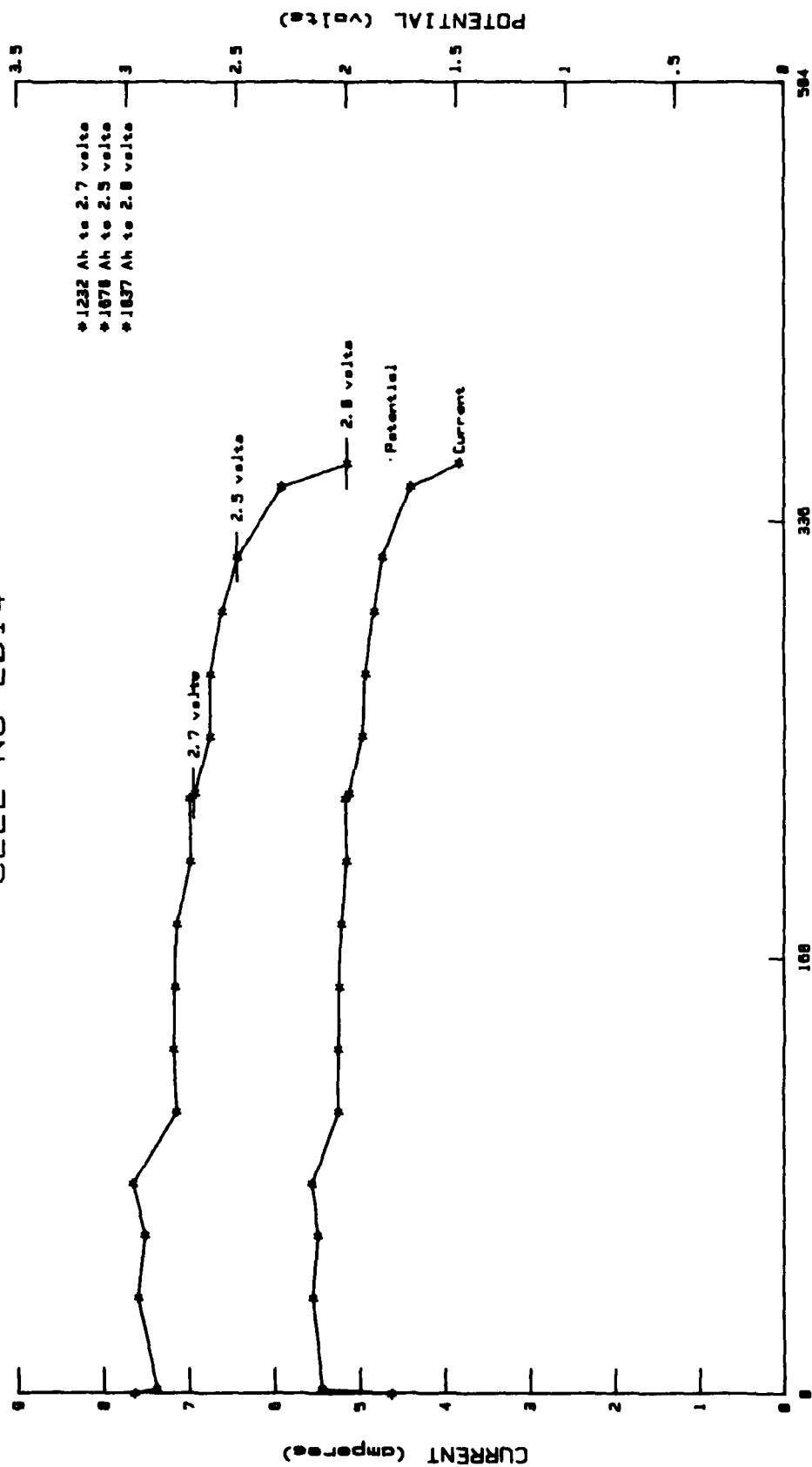


Figure 18

CELL No 2014



CELL No 2015

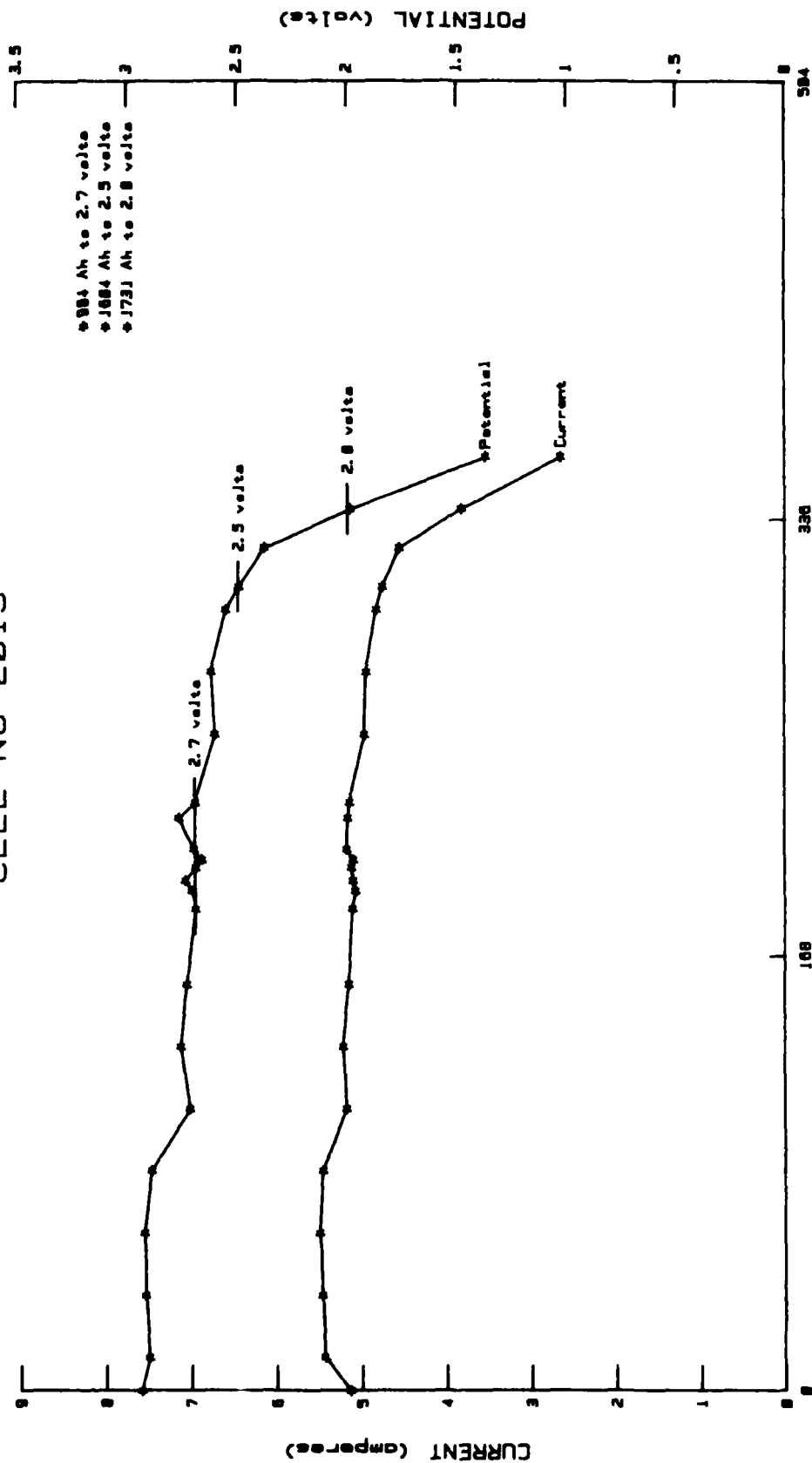


Figure 20

CELL No 2007

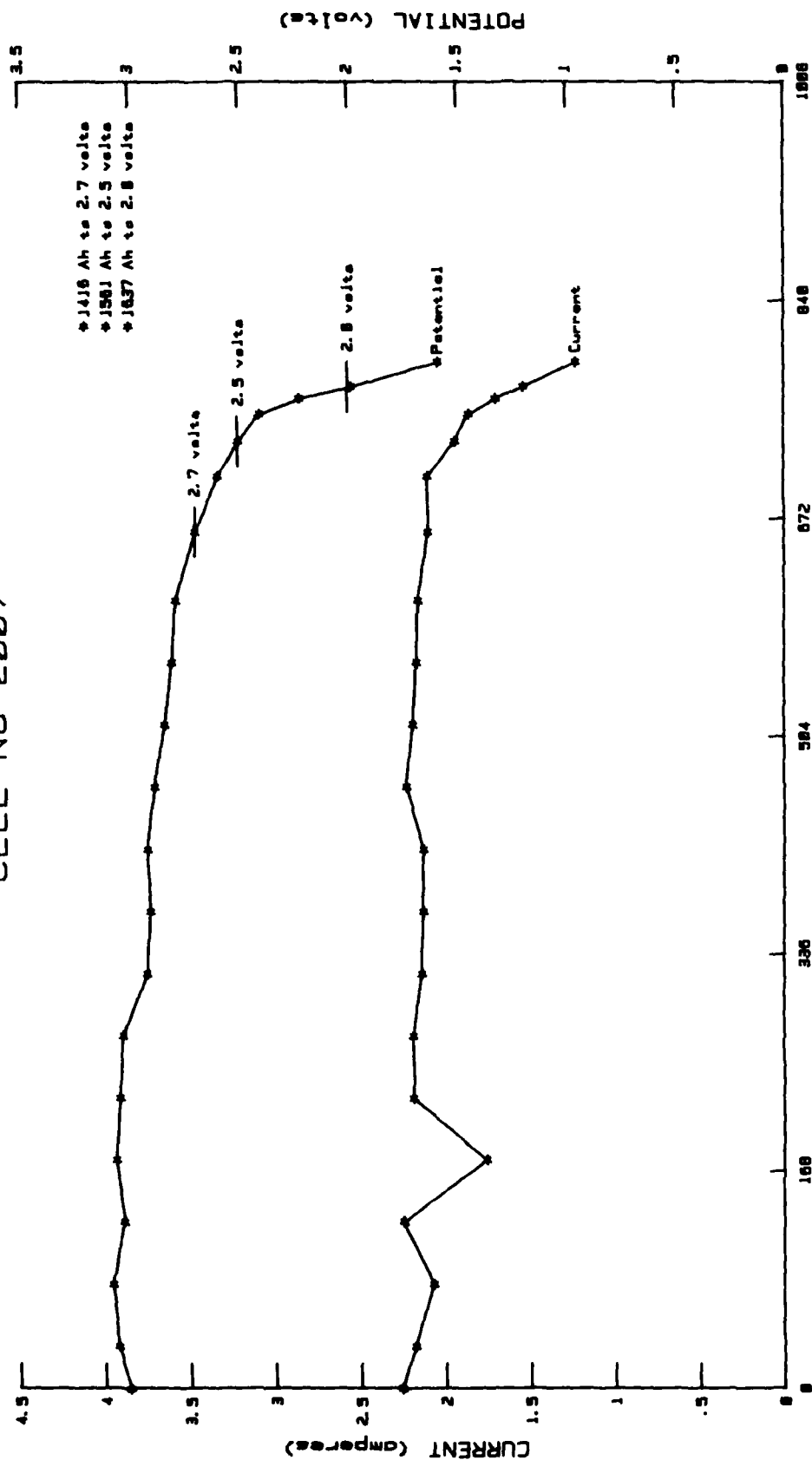


Figure 21

CELL No 2008

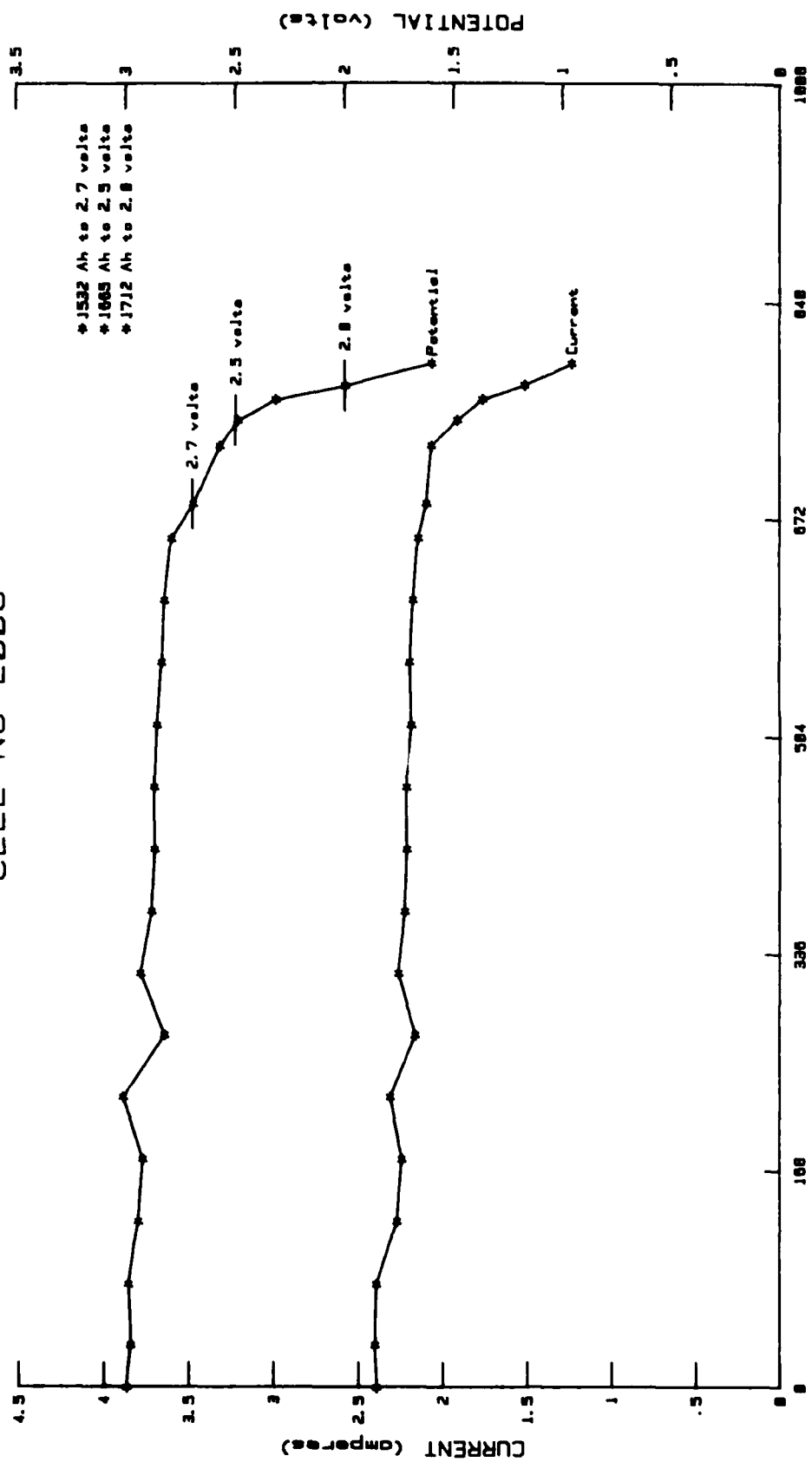


Figure 22

3.0 TESTS AT VARIOUS TEMPERATURES (continued)

Cells numbered 2014 and 2015 from the tests at various discharge rates are used for comparisons since they were discharged at 5.1 amperes. The discharge temperatures of these two cells averaged 18 to 19 Celsius (64-66 Fahrenheit). See Figures 14, 19 and 20.

3.1 5 Celsius Tests

Cells numbered 2013 and 2016 were discharged at an average 4.5 to 4.8 amperes. During the first week of discharge the temperatures of the water baths for these two cells fell to 0 Celsius (32 Fahrenheit) and below. While the cell potentials fell below 2.7 volts on both cells during this time, we reported a later 2.7 volt cutoff time when the water bath temperature was higher. The water baths averaged 8 Celsius (46 Fahrenheit) and the cells ran for 393 to 421 hours for overall capacities above 2.0 volts of 1884 and 1911 ampere-hours. See Figures 14, 23 and 24 for more test data.

3.2 40 Celsius Tests

Cells numbered 2017 and 2018 were discharged at average currents of 5.2 to 5.5 amperes. The heater for the water bath for cell number 2017 failed and allowed the water bath temperature during this time to fall to 10 Celsius (50 Fahrenheit). This lowered the average temperature of the bath to 35 Celsius (95 Fahrenheit). The average bath temperature for number 2018 was 40 Celsius (104 Fahrenheit). Even with this temperature difference the cells performed similar. They ran for 233 and 226 hours and yielded 1327 and 1359 ampere-hours above 2.0 volts. See Figures 14, 25 and 26.

CELL No 2013

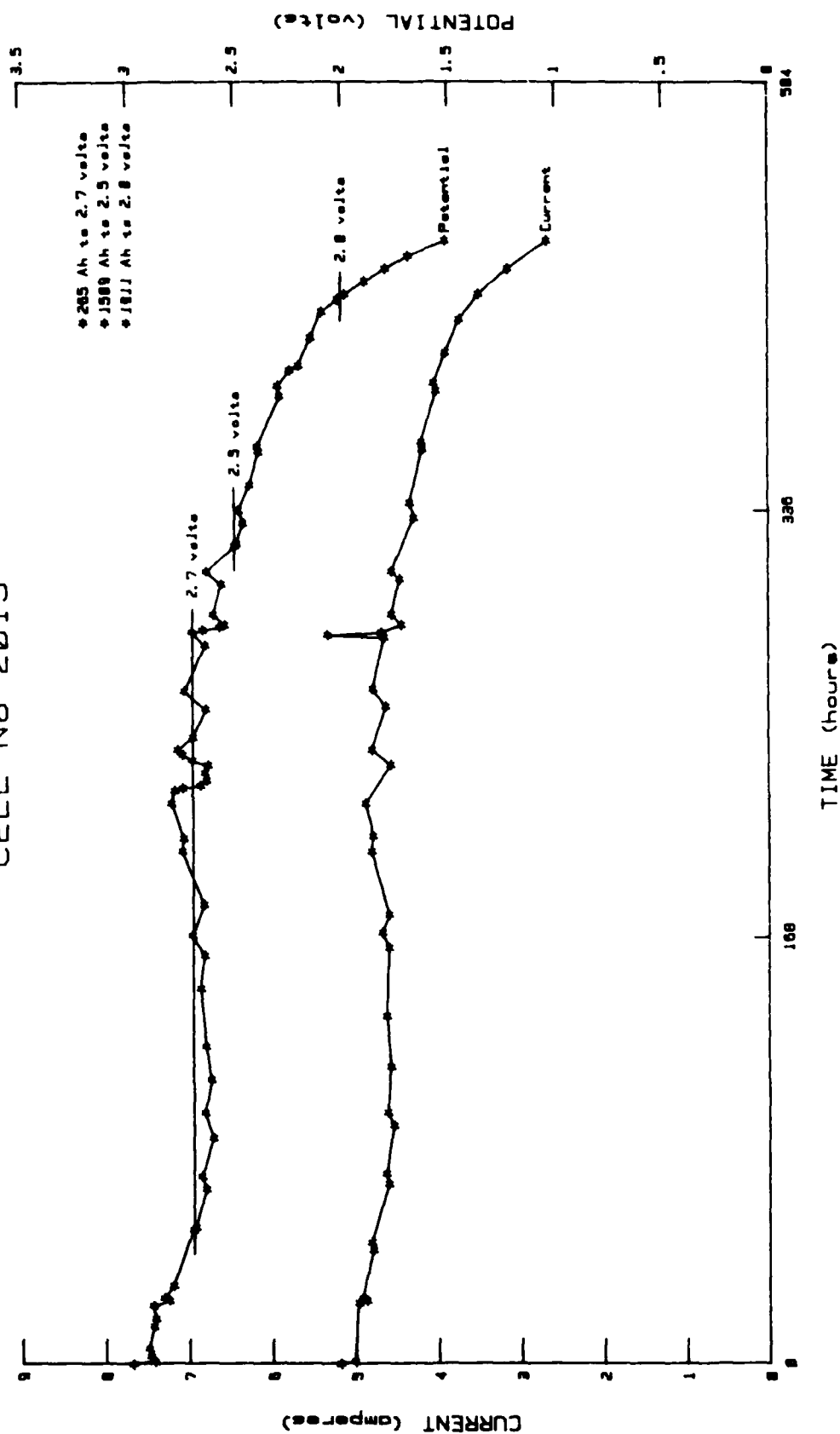


Figure 23

CELL No 2016

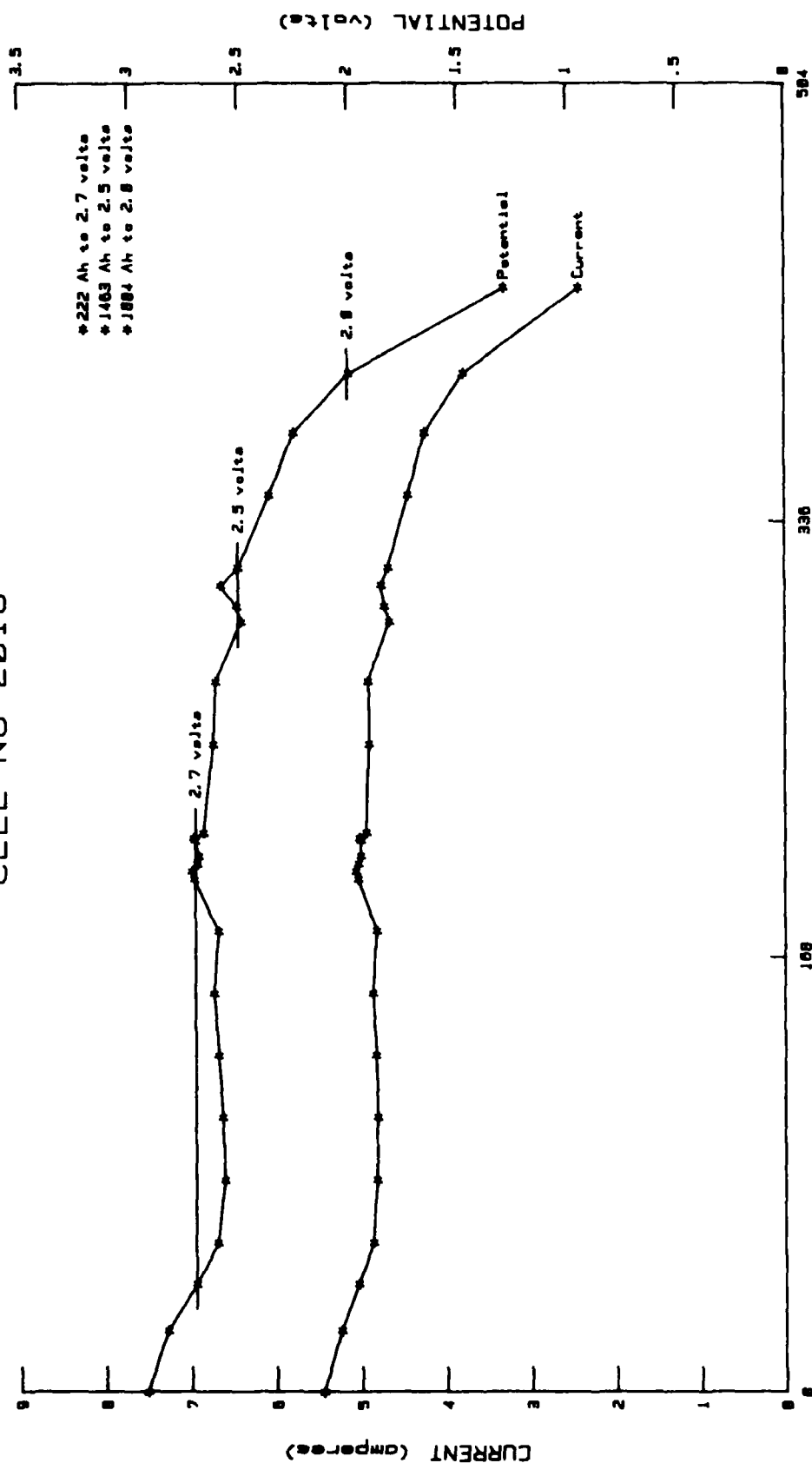


Figure 24

CELL No 2017

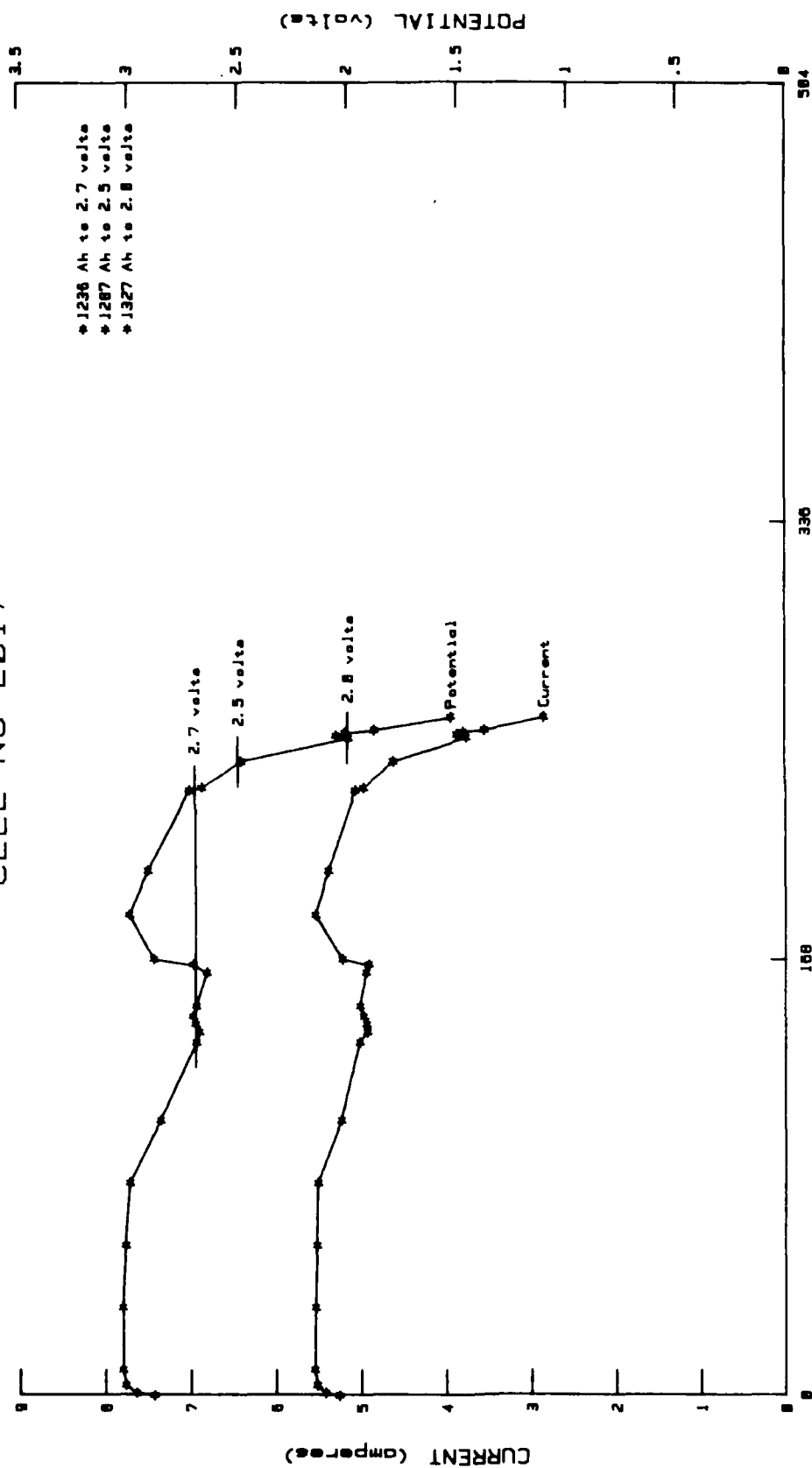


Figure 25

CELL No 2018

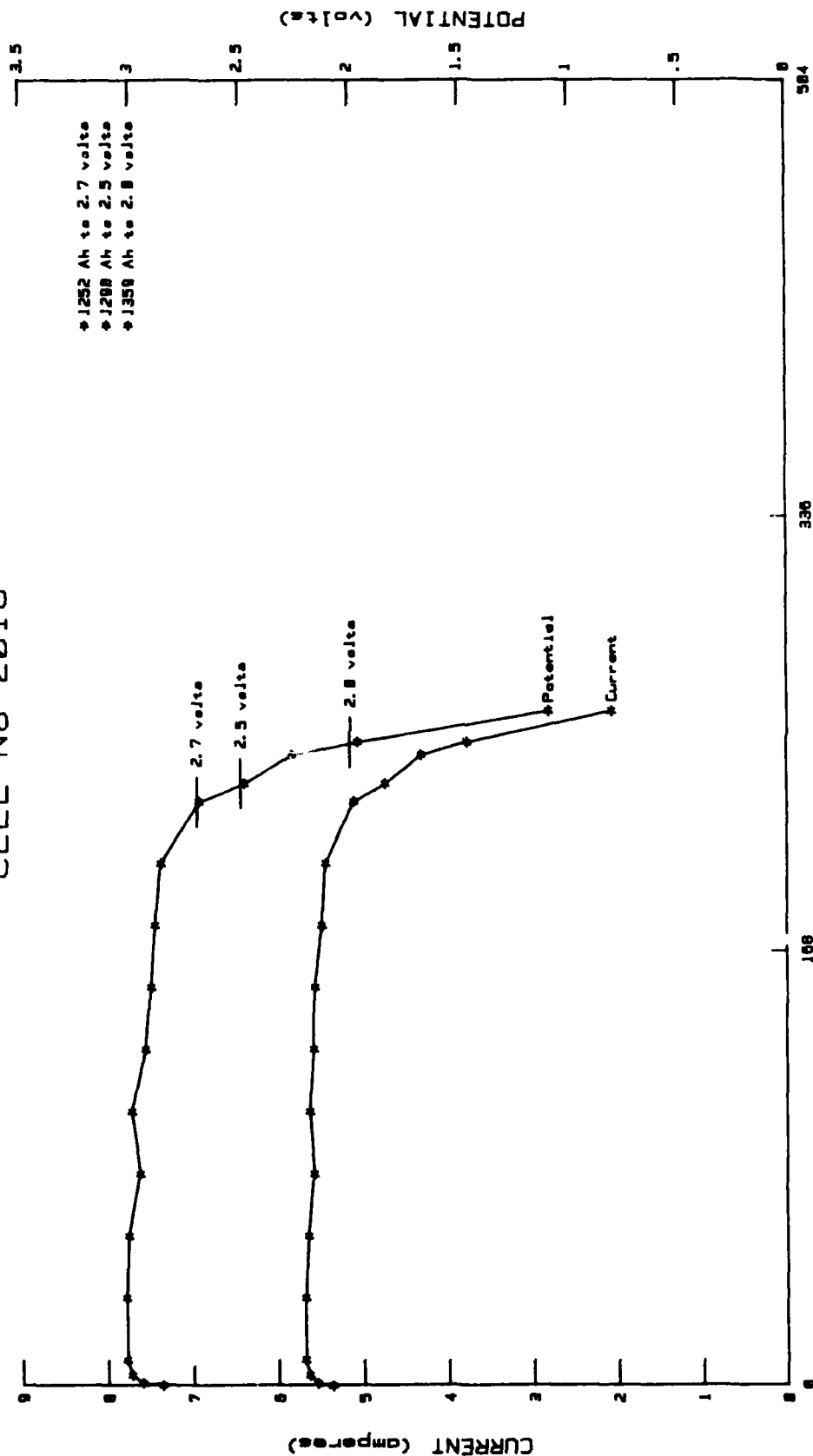


Figure 26

4.0 CONCLUSIONS

A curve showing the energy from the 2000-ampere-hour cells is shown in Figure 27. The points plotted are averages of two cells at each of three rates. The curve representing a 2.0 volt cut off showed no energy loss with rates from 2.1 amperes to 5.1 amperes. It showed an energy decrease of 15% at 9.0 amperes. With a 2.5 volt cut off the curve looked similar, however, the energy loss was greater at higher currents. The energy above 2.5 volts fell 28% when the rate increased from 5.2 amperes to 7.6 amperes. Reduced capacity was expected due to the lower operating potential at these higher rates.

The variable temperature tests showed an interesting pattern. While the higher temperature discharges produced the most energy above a potential of 2.7 volts, the cells favored lower temperatures at lower cut-off potentials (See Figure 28).

VARIABLE CURRENT TESTS

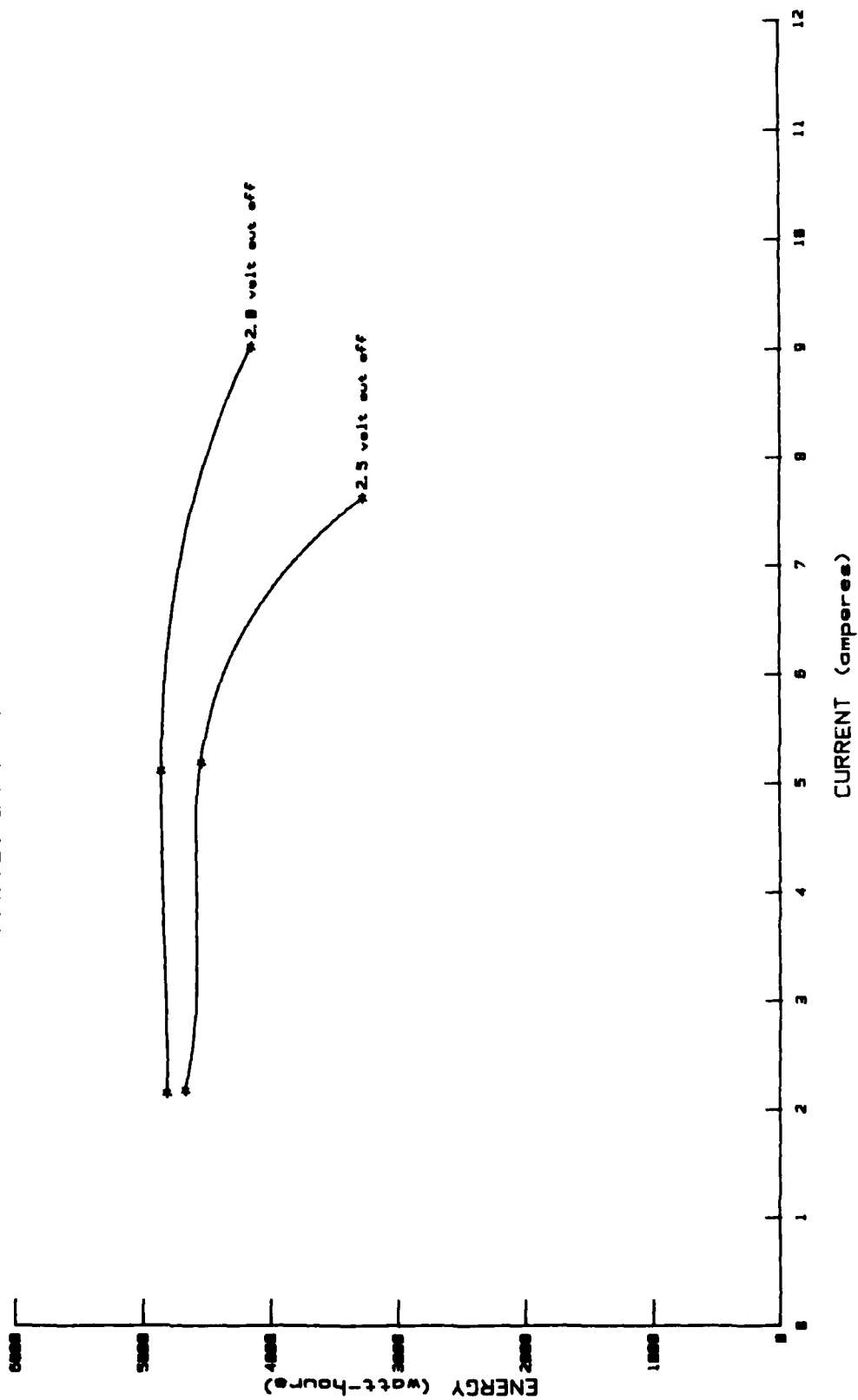


Figure 27

VARIABLE TEMPERATURE TESTS

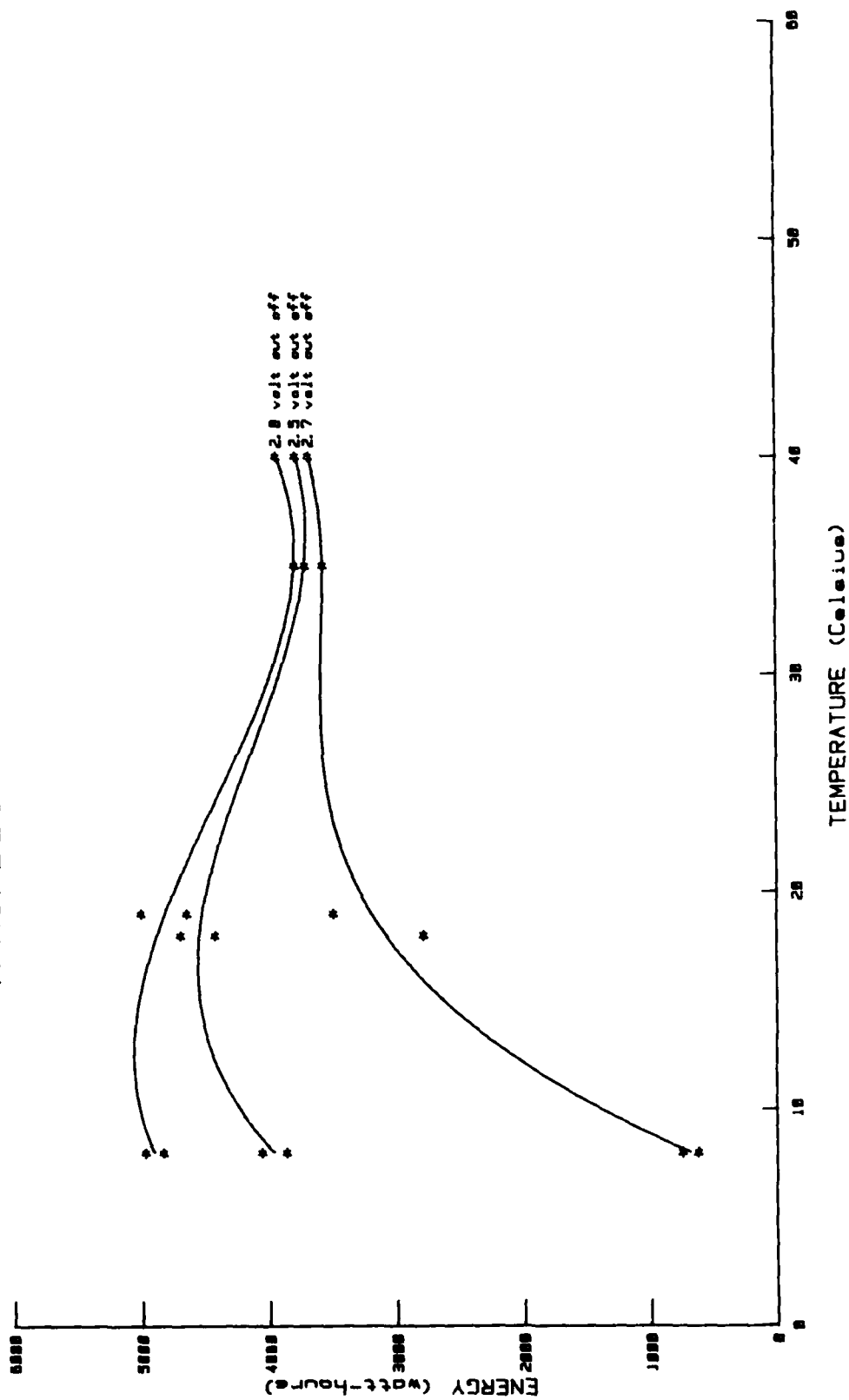


Figure 28

SECTION VII

CELL REVERSAL TEST

(Serial Numbers 2014 and 2015)

Following discharge at the 385 hour rate (reference paragraph 2.3 of Section VI), the subject cells were reverse discharged with power supplies at the 385 hour rate (5.2 amperes) to an intended 100% reversal (2000 ampere-hours). However, after 54 hours (approximately 180 ampere-hours), the current of Cell Number 2014 decayed to zero and after 53 hours (approximately 145 ampere-hours) the current of Cell Number 2015 decayed to zero. This decay was due to the cell potential matching the maximum potential of our 40 volt power supplies. Neither cell would accept any further discharge. The test was terminated.

Cell Number 2014 indicated a beginning pressure of 1.2 atmospheres (17 pounds per square inch) and after 22 hours, the pressure had risen to 2.1 atmospheres (30 pounds per square inch). After 48 hours, a pressure reading was taken and found to be zero. Apparently venting had occurred.

Cell Number 2014's temperature rose from approximately 26 Celsius (70 Fahrenheit) to 50 Celsius (126 Fahrenheit) from 52 to 54 hours. See Figures 29, 30, 31, and 32 for additional information.

CELL No 2014 REVERSAL

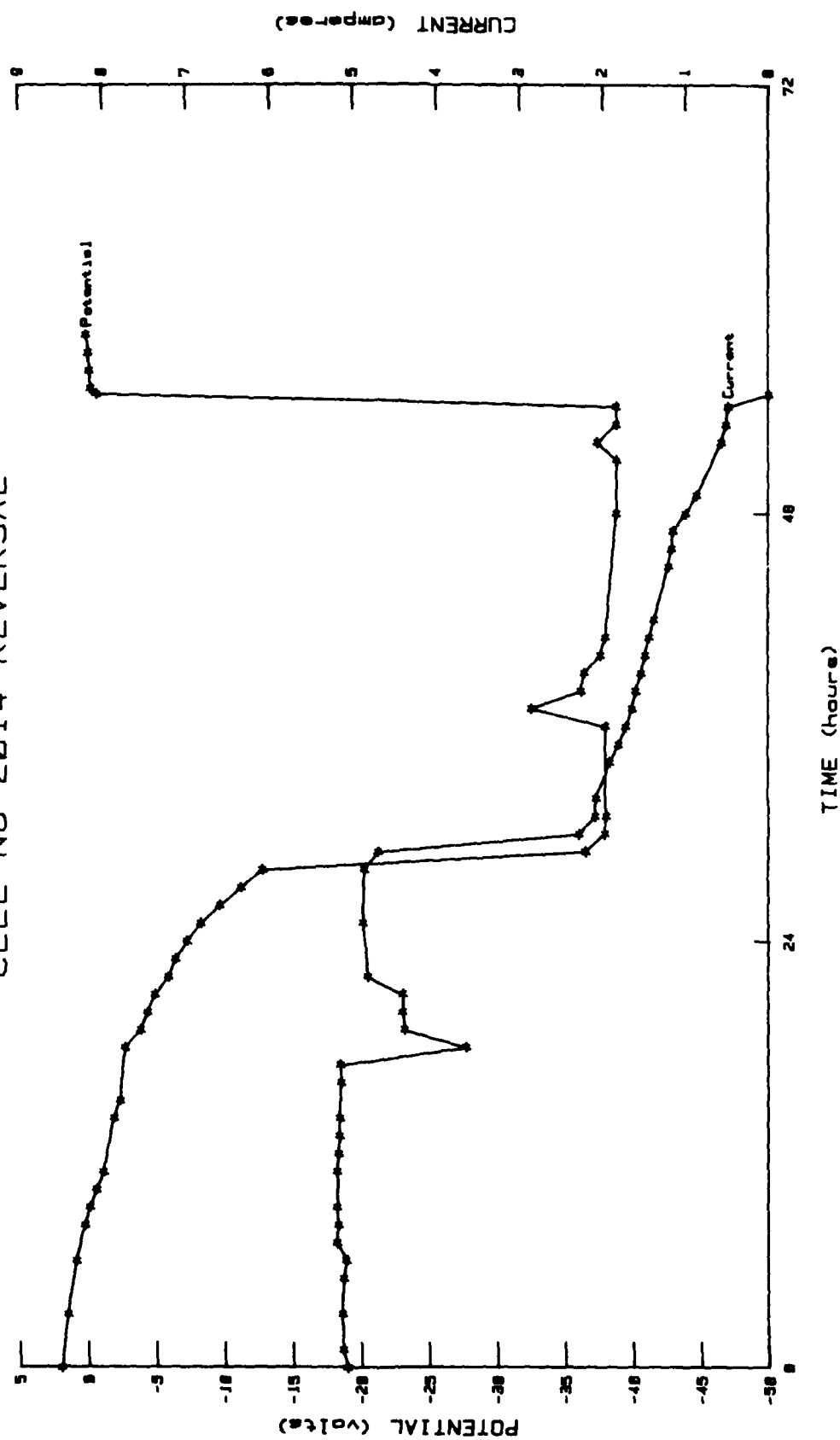


Figure 29

CELL No 2014 REVERSAL

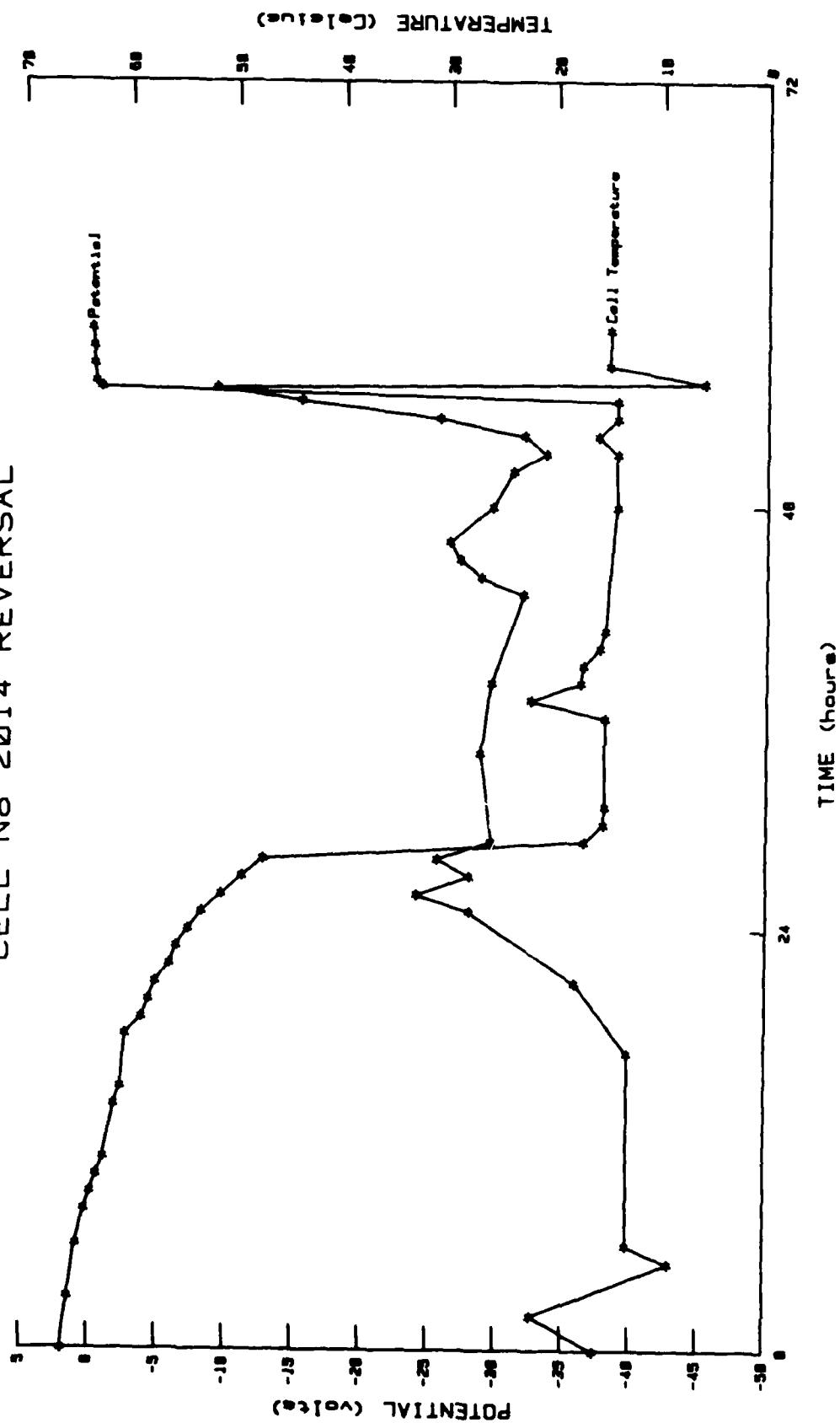


Figure 30

CELL No 2015 REVERSAL

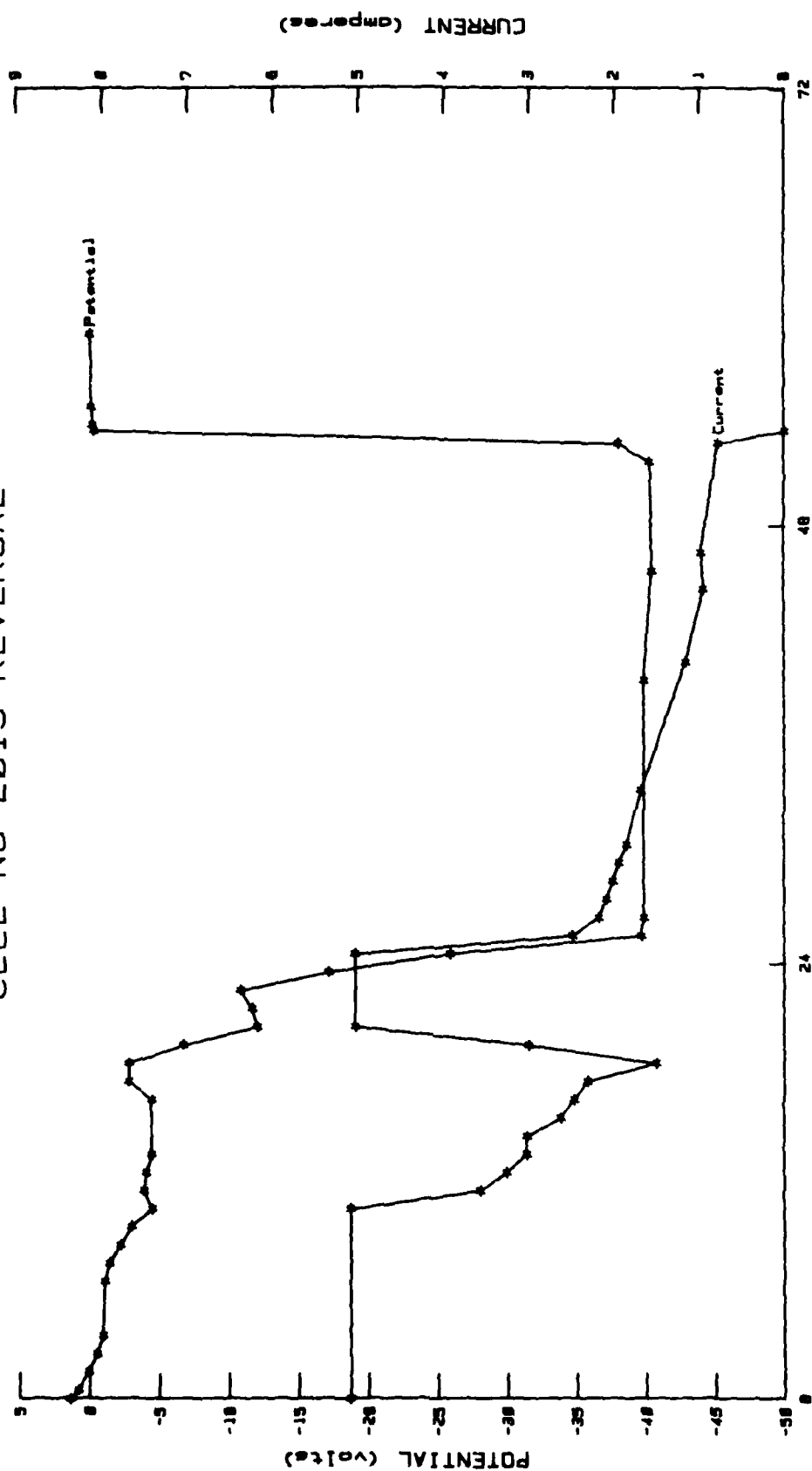


Figure 31

CELL No 2015 REVERSAL

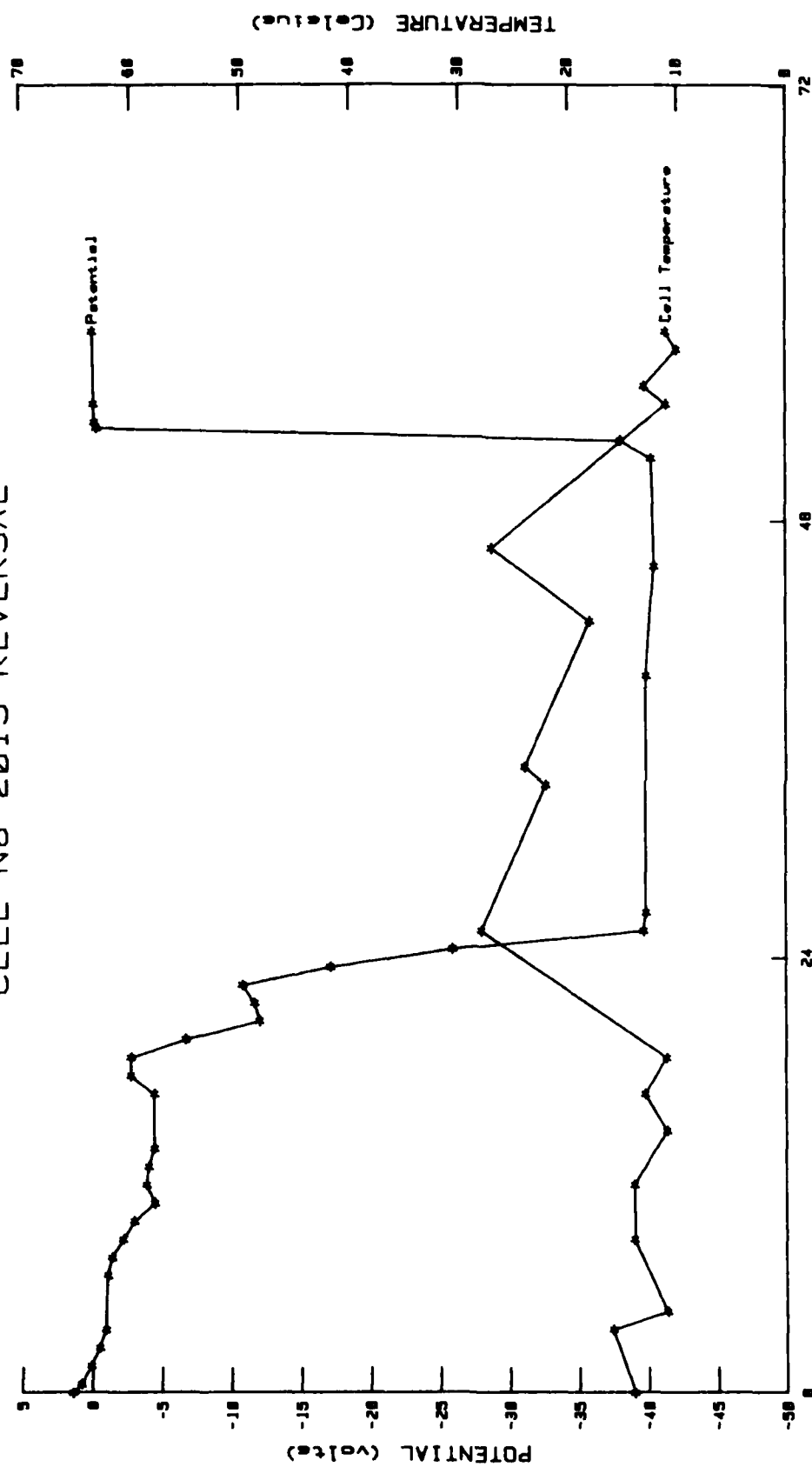


Figure 32

SECTION VIII
EXTENDED STAND AND INTERMITTENT DISCHARGE TESTS
(200 Ampere-hour Cells)

1.0 INTRODUCTION

Calculations indicated that about 0.65 liters (0.17 gallons) electrolyte were required to fill the voids in the cathodes and separators and wet the cell pack in the 200-ampere-hour cells. Calculations also indicated that a volume of 0.90 liters (0.24 gallons) would completely fill the cell, leaving no room for expansion. In order to eliminate the possibility of cell case or vent diaphragm rupture, a volume of 0.80 liters (0.21 gallons) of electrolyte was selected for the 200-ampere-hour cells. This permitted an adequate safety margin.

Eight cells were discharged at varying rates and intervals according to the following schedule:

- 1) cells numbered 206 and 207 were pulsed at a 0.5 ampere rate at various intervals. After 42 days the cells were continuously discharged at 0.5 amperes.
- 2) Cells numbered 204 and 205 were step-loaded from 0.26 - 1.625 amperes.
- 3) Cells numbered 206 and 207 were pulsed at a 0.52 ampere rate at various intervals. After 42 days the cells were continuously discharged at 0.5 amperes.
- 4) Cells numbered 208 and 209 were discharged at 0.02 amperes for 40 days and then discharged at 0.5 amperes.

These cells were resistive discharged using the automatic discharge controller mentioned in Section V-2.0.

1.0 INTRODUCTION (continued)

Cell Serial Numbers 202 through 205 were placed in a common rack and separated from each other by an insulated metal spacer and a space for water flow between each cell. This permitted a common water bath for those cells. Cell Serial Number 206 through 209 were also racked commonly as described for Cells 202 through 205 and placed in a separate water bath.

Each block of cells was activated by evacuating all four cells through a common manifold connected to the vacuum supply. This permitted equal evacuation of all cells. Each cell was supplied with individual bottles of electrolyte which were connected to the respective cells through a three-way valve. Following activation, the system was closed, and the cells were allowed to stand a day to assure proper wetting of the cell stack.

2.0 TEST RESULTS

2.1 Cell Numbers 202 and 203

Following 411 hours at open circuit, the cells were loaded and discharged to 2.00 volts. Cell Number 202 was discharged at an average load of 0.495 amperes and yielded 209 ampere-hours; while Cell Number 203 was discharged at an average rate of 0.517 amperes with a capacity yield of 235 ampere-hours. After 419 hours of discharge, Cell Number 202 was loaded at approximately 0.7 amperes. See Figures 33, 34, and 35 of this report for additional information.

RESULTS OF EXTENDED STAND AND
INTERMITTANT DISCHARGE TESTS

CELL NUMBER	ELECTROLYTE VOLUME L.(GAL)	DIS.CUR AMPERES	WATER BATH TEMPERATURE CELSIUS(F)	* DISCHARGE TIME HOURS	* ENERGY CAPACITY WATT- HOURS	* ENER. WH/ DM ³	* DENS.* WH/ IN ³	* SPEC. WH/ KG.	* ENER. WH/ LB.	* CUT-OFF POTENTIAL VOLTS
202	.80(.21)	.495	10 (50)	422	209	576	7.56	230	104	2.00
203	.80(.21)	.517	10 (50)	453	235	642	8.42	257	117	2.00
204	.80(.21)	1.290	12 (54)	163	210	549	7.20	220	100	2.00
205	.80(.21)	1.295	12 (54)	163	211	552	7.23	221	100	2.00
206	.55(.15)	.493	13 (55)	378	209	550	7.21	265	120	2.00
207	.80(.21)	.496	13 (55)	449	234	628	8.23	251	114	2.00
208	.80(.21)	.164	13 (55)	1406	230	616	8.08	246	112	2.00
209	.80(.21)	.158	13 (55)	1384	219	586	7.69	234	106	2.00

Figure 33

CELL No 202

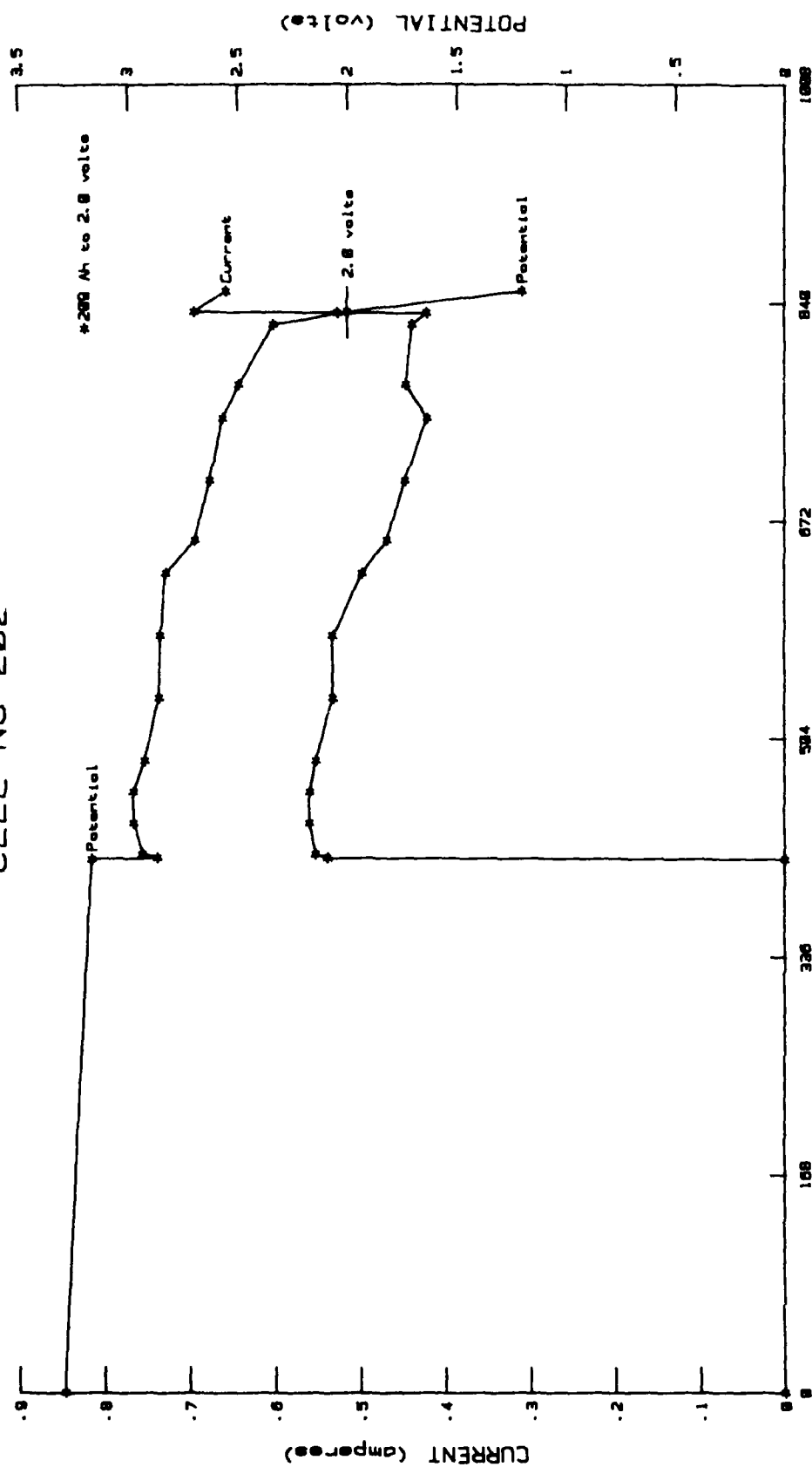


Figure 34

CELL No 203

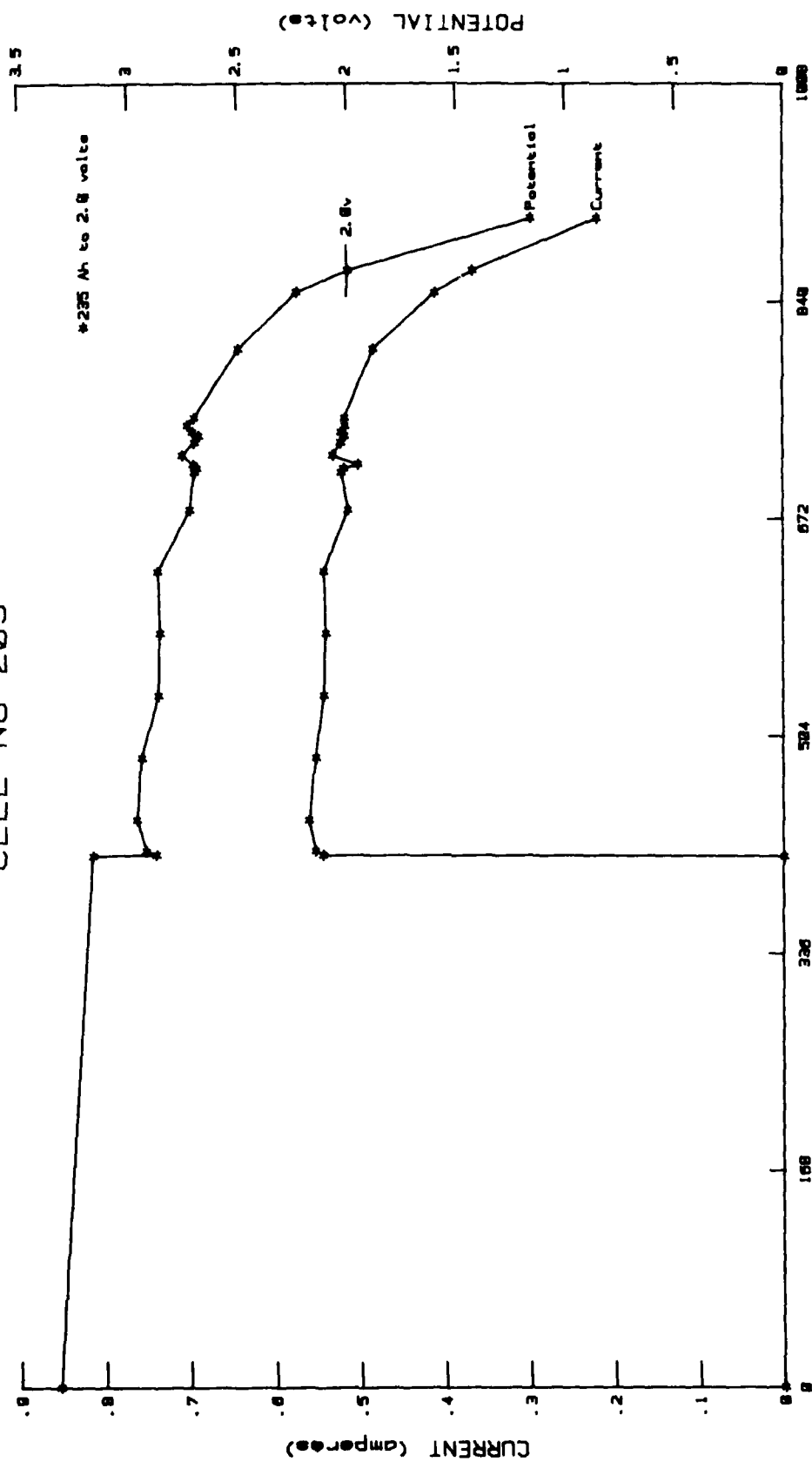


Figure 35

2.0 TEST RESULTS (continued)

2.2 Cell Numbers 204 and 205

Cells 204 and 205 were loaded at intermittent rates of 0.26, 0.52, 1.04, and 1.625 amperes. Following intermittent loading, the cells were discharged at a steady rate of 1.625 amperes.

Cell Number 204 yielded 210 ampere-hours to 2.00 volts and Cell Number 205 yielded 211 ampere-hours to 2.00 volts. After the completion of discharge as described above, the cells were permitted to stand at open circuit for a period of 20 hours. The cells were then reloaded at 0.26 and 0.52 amperes to 2.00 volts. An additional 21 and 22 ampere-hours, respectively, were obtained from the cells.

See Figures 33, 36 and 37 for test data.

2.3 Cell Numbers 206 and 207

Cells 206 and 207 were activated as described and allowed to stand at open circuit for a period of 27 hours to assure proper wetting. Cell Number 206 received only 0.55 liters (0.15 gallons) of electrolyte.

The cells were pulse loaded at varying intervals at a 0.52 ampere rate. After 1,018 hours of intermittent discharges, the cells were continuously discharged at an average of 0.493 and 0.496 amperes. The capacity of Cell 206 to 2.00 volts was 209 ampere-hours and the capacity of Cell 207 to 2.00 volts was 234 ampere-hours. Apparently the difference in yielded capacity is reflected in the quantities of electrolyte received by each cell. See Figures 33, 38, and 39 for test data.

CELL No 204

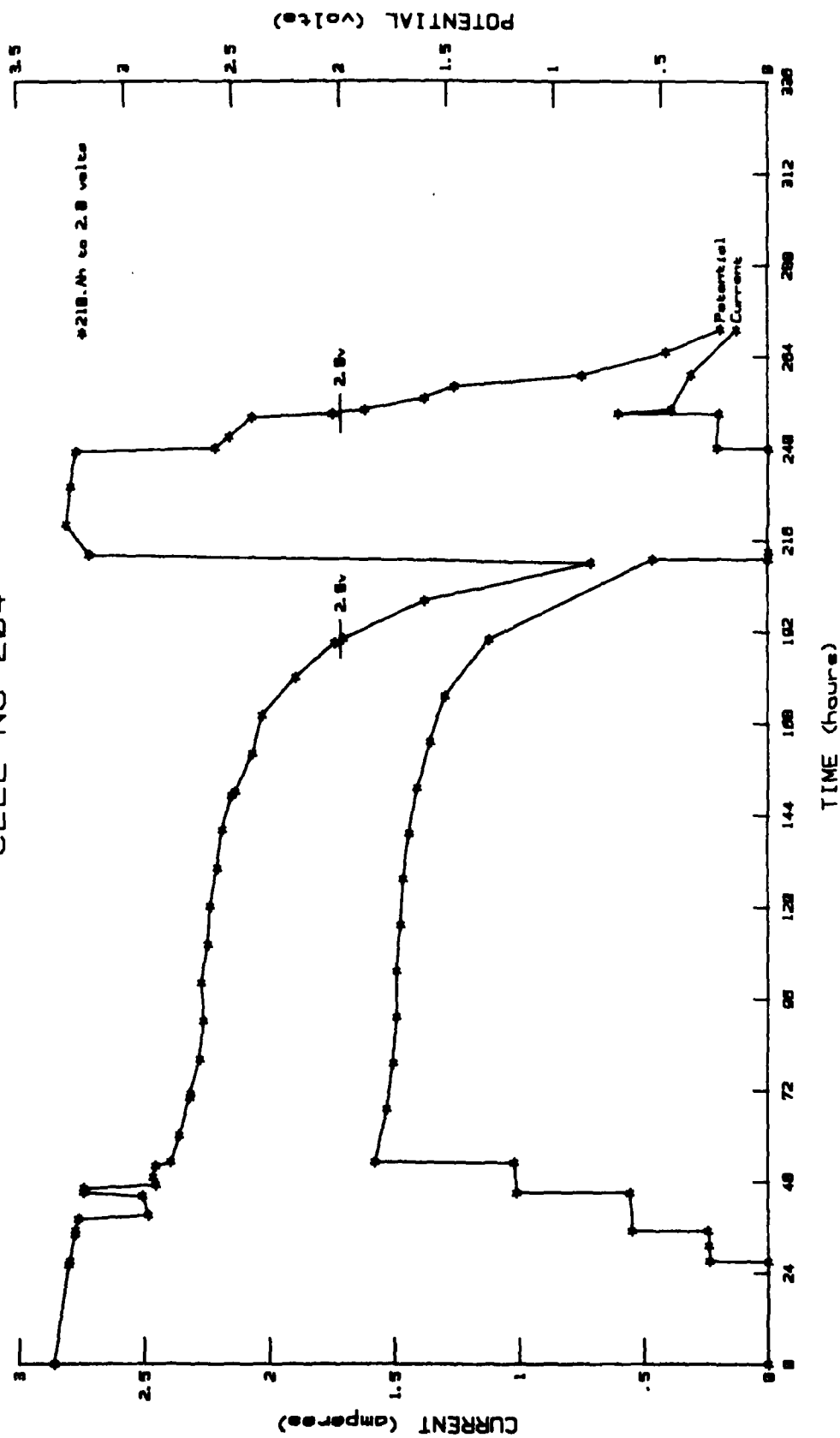


Figure 36

CELL No 205

POTENTIAL (volts)

CURRENT (amperes)

TIME (hours)

2.8V

2.8V

Potential

Current

Figure 37

CELL No 206

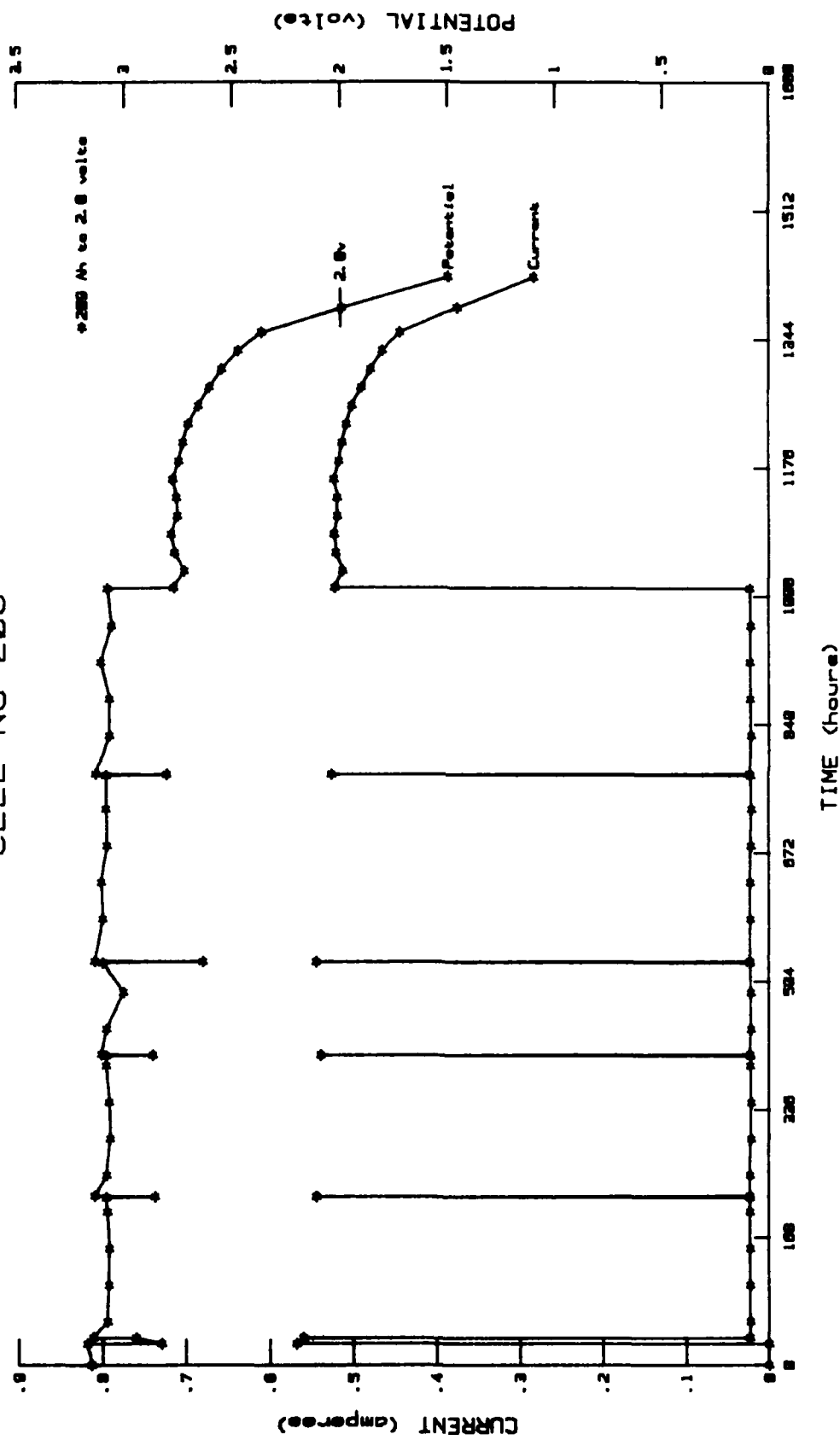


Figure 38

CELL No 207

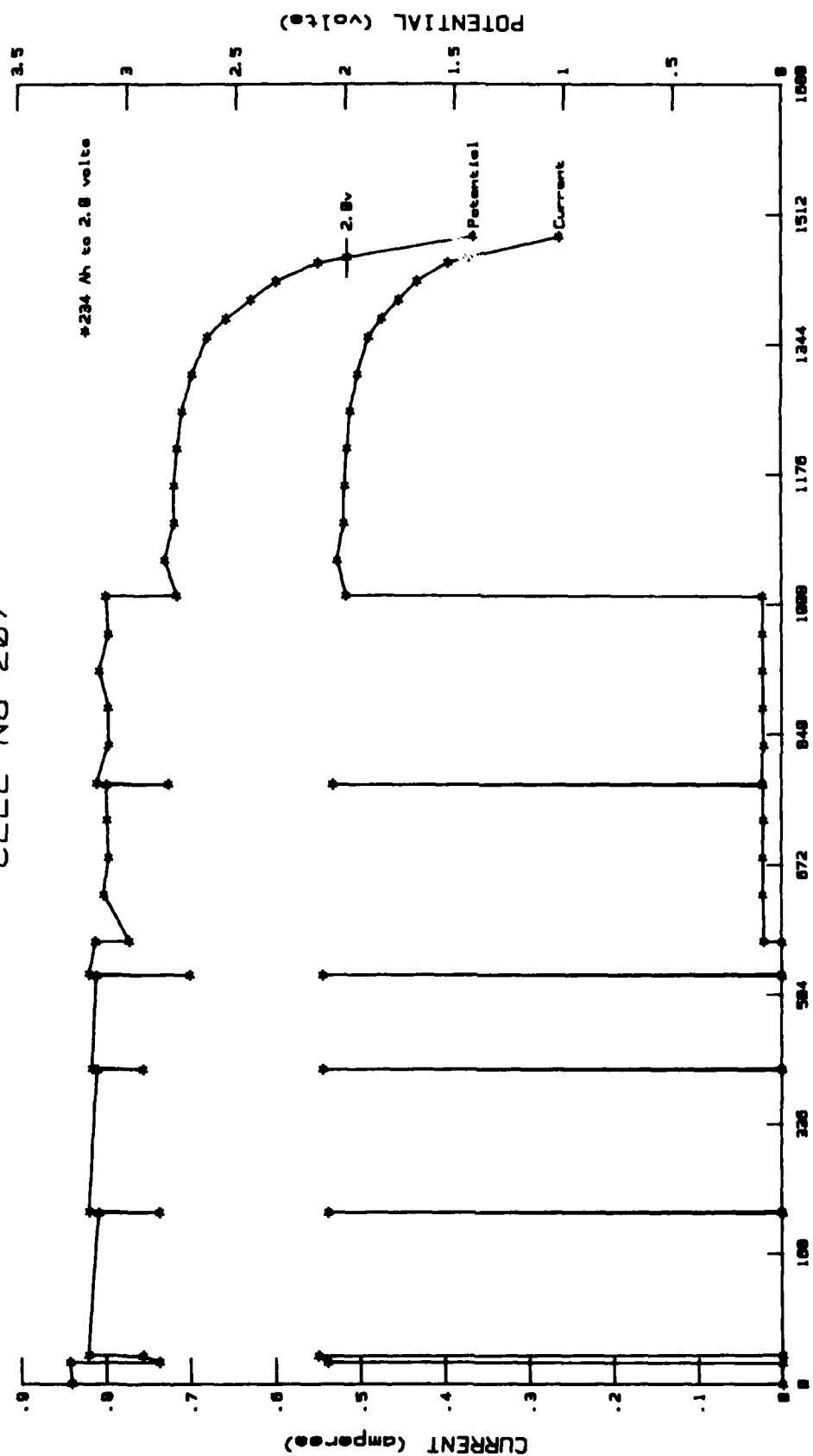


Figure 39

2.4 Cell Numbers 208 and 209

Cells 208 and 209 were loaded at 0.022 amperes for a period of 955 hours. At 955 hours the rates on the cells were increased to an average of 0.49 amperes and the discharge continued to cell depletion.

The capacity yield of Cell 208 was 230 ampere-hours to 2.00 volts, and the capacity yield of Cell 209 was 219 ampere-hours to 2.00 volts.

3.0 DISCUSSION

Lithium/thionyl chloride cells show an accelerated loss of capacity when discharged intermittently or at very low rates. The cells discharged in this test did not show this accelerated capacity loss. See Figure 42. The points at the left end of the curve represent cells 204 and 205 which were discharged in 163 hours. The lower capacity of these cells can be explained by the higher currents (1.3 amperes). The cells were loaded a second time and yielded 20 ampere-hours more capacity at the 0.5 ampere rate. If this additional capacity were added to the shown totals the curve on Figure 42 would have a very slight negative slope.

The 200-ampere-hour cells performed remarkably better than the 2000 ampere-hour cells. While the energy density and the specific energy are similar to the better performances of the larger cells, the cathode utilization in the smaller cells is far better. Cathode utilization ranged from 0.35 to 0.40 ampere-hours per cubic centimeter (5.8 to 6.5 ampere hours per cubic inch) in the small cells. Cathode utilization in the large cells ranged from 0.16 to 0.33 ampere-hours per cubic centimeter (2.6 to 5.3 ampere-hours per cubic inch).

CELL No 208

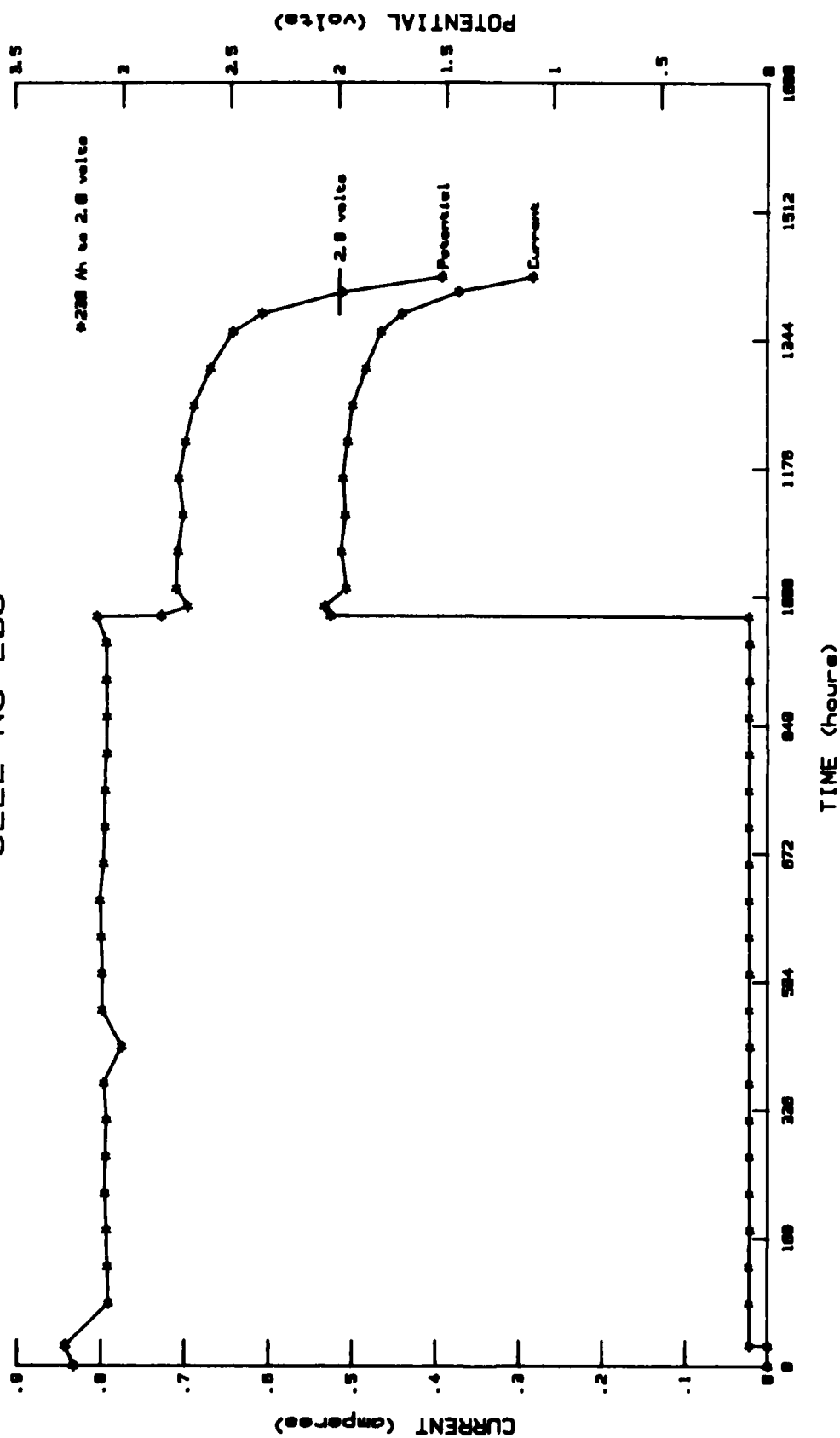


Figure 40

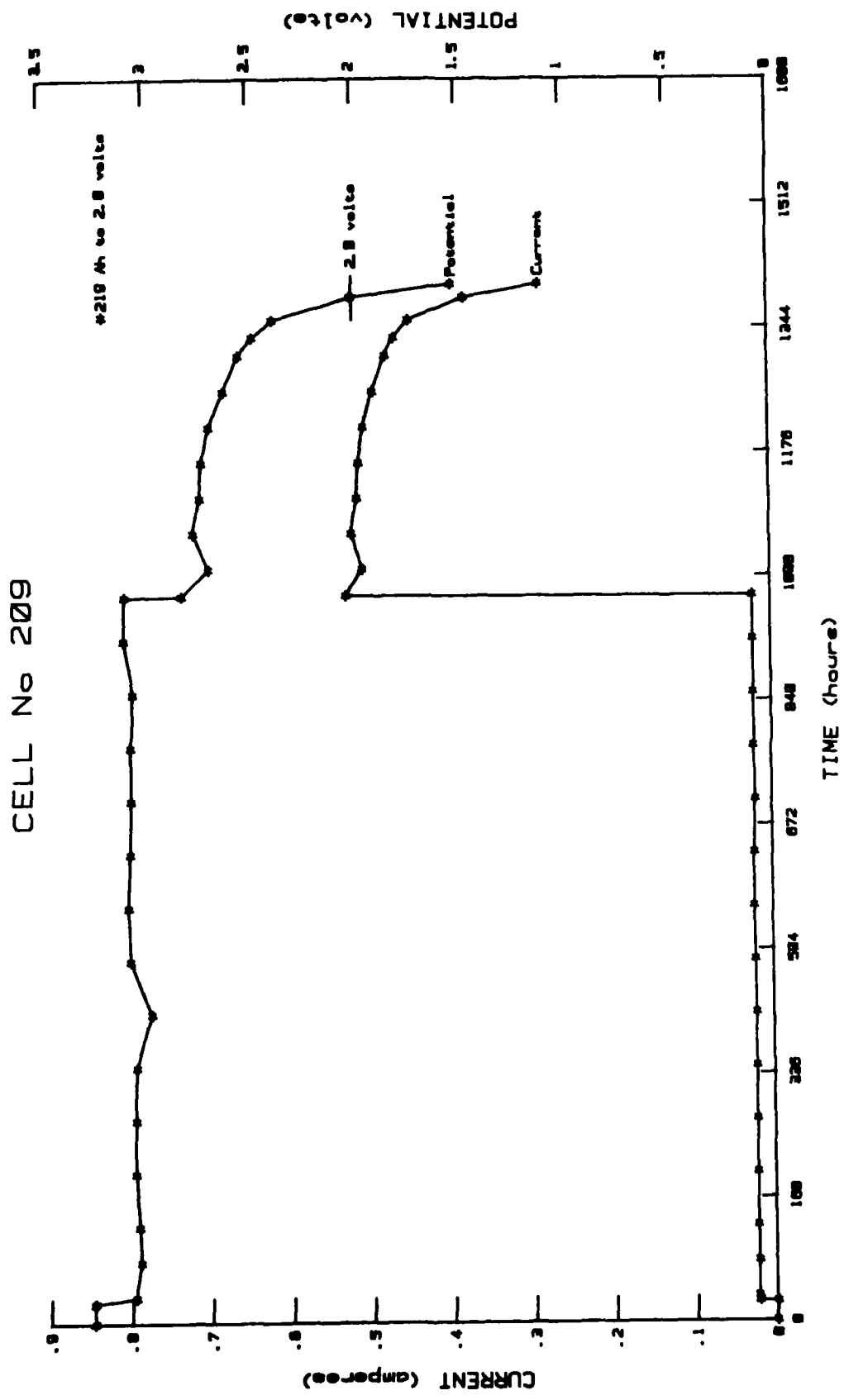


Figure 41

3.0 DISCUSSION (continued)

The highest current density tested on the large cells was 0.93 milliamperes per square centimeter (6.0 milliamperes per square inch) and that cell produced only 0.24 ampere-hours per cubic centimeter (4.0 ampere-hours per cubic inch) of cathode. A 200-ampere-hour cell was discharged at a peak 0.85 milliamperes per square centimeter (5.5 milliamperes per square inch) and produced 0.36 ampere-hours per cubic centimeter of cathode.

LONG TERM DISCHARGES

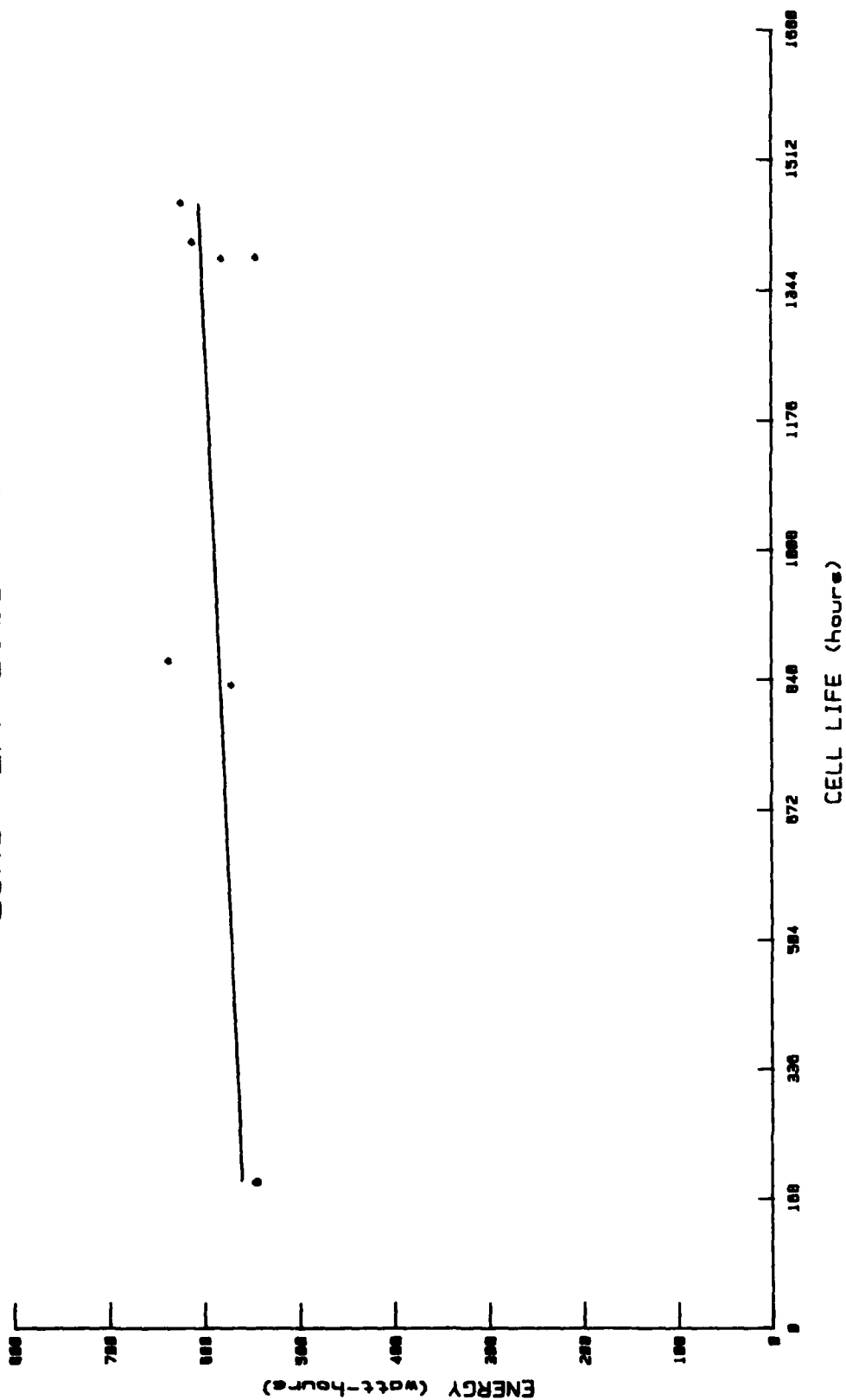


Figure 42

SECTION IX

SUMMARY

Performance variances between the 2000-ampere-hour and the 200-ampere hour cells did exist. The 200-ampere-hour cells performed much better from a cathode utilization standpoint. Cathode utilization ranged from 0.16-0.33 ampere hour per cubic centimeter (2.6 to 5.3 ampere hours per cubic inch) for the 2000-ampere-hour cells versus 0.35 to 0.40 ampere hours per cubic centimeter (5.8 to 6.5 ampere hours per cubic inch) for the 200-ampere-hour cells.

Had the 2000-ampere-hour cells performed equal to the 200-ampere-hour cells in relation to the cathode utilization, the 2000-ampere-hour cells would have yielded from 2310-ampere-hours and 6040 watt-hours at an average 14.2 amperes to 2574 ampere-hours and 6910 watt-hours at 5.46 amperes. The 2000-ampere-hour cells would have yielded as much as 610 watt-hours per cubic decimeter (10 watt-hours per cubic inch) and 350 watt-hours per kilogram (160 watt-hours per pound.)

The cathodes, anodes and separators were of identical construction for both the 2000 and the 200-ampere-hour cells, with the only difference being the number of components used. Several assumptions can be made as to the cause of the variation in performance attained, for example:

- 1) Differences in vacuum obtainable during activation due to the larger number of void spaces between the electrodes and the collapsibility of the large cell case into these void areas. This collapsibility of the larger cell case prevented evacuation of the air in the cell adequate to provide complete and thorough wetting of the electrodes near the middle of the cell.

SUMMARY (continued)

2) Temperature near the middle of the larger cell became sufficiently high to vaporize the electrolyte and cause dry spots and subsequent loss of active surface area of the electrodes near the middle of the cell. This higher temperature is probably caused by a combination of things:

- a) Inadequate thermal conductance of the anodes, cathodes, separators and stainless steel cell case retarded the heat flow from the center of the cell.
- b) As the cell dries out near the center and the surface area decreases the current density increases, thus causing additional increases in heat.

Eagle Picher would recommend that any future cell design incorporate measures to prevent these problem areas. Some things that should be examined are:

- 1) Stiffer cell case walls (ribbed, etc) to prevent collapsibility during activation or activation of the cell in a vacuum box to prevent atmospheric pressure from collapsing the cell.
- 2) Design of the cell in a manner that would permit heat measurement near the middle of the cell pack. This would, by necessity, take into consideration the corrosiveness of the thionyl chloride electrolyte and should provide a method of isolation from the electrolyte.

Other areas of future study should be: Increasing specific energy and energy densities through anode stability studies, better cathode matrix utilization and further, more refined electrolyte studies.

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